WL-TR-92-4027

EVALUATION OF CHARACTERIZATION TECHNIQUES FOR CARBON-CARBON COMPOSITES

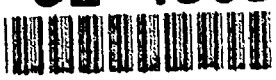
William Ragland
University of Dayton Research Institute
300 College Park Avenue
Dayton, OH 45469-0168

MAY 1992

Interim Report for the Period June 1990 - June 1991

Approved for public release; distribution unlimited.

MATERIALS DIRECTORATE
WRIGHT LABORATORY
AIR FORCE SYSTEMS COMMAND
WRIGHT-PATTERSON AIR FORCE BASE, OH 45433-6533



NOTICE

When government drawings, specifications, or other data are used for any purpose other than in connection with a definitely Government-related procurement, the United States Government incurs no responsibility or any obligation whatsoever. The fact that the Government may have formulated or in any way supplied the said drawings, specifications, or other data, is not to be regarded by implication, or otherwise in any manner construed, as licensing the holder, or any other person or corporation; or as conveying any rights or permission to manufacture, use, or sell any patented invention that may in any way be related thereto.

This report is releasable to the National Technical Information Service (NTIS). At NTIS, it will be available to the general public, including foreign nations.

This technical report has been reviewed and is approved for publication.

Kenneth M. Johnson

KENNETH M. JOHNSON, Mat'ls Engr
Composites Group
Structural Materials Branch

CHARLES E. BROWNING, Chief
Structural Materials Branch
Nonmetallic Materials Division

FOR THE COMMANDER

MERRILL L. MINGES, Director
Nonmetallic Materials Division
Materials Directorate

If your address has changed, if you wish to be removed from our mailing list, or if the addressee is no longer employed by your organization, please notify WL/MLBC, Wright-Patterson AFB, OH 45433-6533 to help maintain a current mailing list.

Copies of this report should not be returned unless return is required by security considerations, contractual obligations, or notice on a specific document.

REPORT DOCUMENTATION PAGE

Form Approved
OMB No. 0704-0188

1. AGENCY USE ONLY (Leave blank)	2. REPORT DATE	3. REPORT TYPE AND DATES COVERED
	May 1992	Interim Report - June 90-June 91

4. TITLE AND SUBTITLE
EVALUATION OF CHARACTERIZATION TECHNIQUES FOR CARBON-CARBON COMPOSITES

5. FUNDING NUMBERS
C-F33615-91-C-5618
PE-62102F
PR-2419
TA-01
WU-G1

6. AUTHOR(S)
William Ragland

7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES)
University of Dayton Research Institute
300 College Park Avenue
Dayton, OH 45469-0168

8. PERFORMING ORGANIZATION REPORT NUMBER
UDR-TR-92-25

9. SPONSORING/MONITORING AGENCY NAME(S) AND ADDRESS(ES)
Kenneth M. Johnson/(513)255-9073
Materials Directorate (WL/MLBC)
Wright Laboratory
Wright-Patterson AFB, OH 45433-6533

10. SPONSORING/MONITORING AGENCY REPORT NUMBER
WL-TR-92-4027

11. SUPPLEMENTARY NOTES

12a. DISTRIBUTION/AVAILABILITY STATEMENT
Approved for public release; distribution is unlimited.

12b. DISTRIBUTION CODE

13. ABSTRACT *(Maximum 200 words)*

Characterization techniques for organic/inorganic composites are well established and documented; however, this is not the case regarding carbon-carbon composites.

Due to expansion of the Materials Directorate in-house carbon-carbon composite research effort, the knowledge to characterize these complex composite structures is essential to their development.

This report describes both existing techniques and techniques developed in the Materials Directorate to characterize carbon-carbon composites.

14. SUBJECT TERMS
carbon-carbon composites; computer tomography; microstructure characterization techniques; vacuum impregnation; sectioning nondestructive evaluation; optical microscopy

15. NUMBER OF PAGES
83

16. PRICE CODE

17. SECURITY CLASSIFICATION OF REPORT	18. SECURITY CLASSIFICATION OF THIS PAGE	19. SECURITY CLASSIFICATION OF ABSTRACT	20. LIMITATION OF ABSTRACT
Unclassified	Unclassified	Unclassified	SAR

TABLE OF CONTENTS

TABLE OF CONTENTS (Concluded)

LIST OF ILLUSTRATIONS

LIST OF ILLUSTRATIONS (Continued)

LIST OF ILLUSTRATIONS (Continued)

LIST OF ILLUSTRATIONS (Concluded)

LIST OF TABLES

FOREWORD

This report was prepared by the University of Dayton Research Institute under Air Force Contract No. F33615-91-C-5618, Project No. 2419, Task No. 241902. The work was administered under the direction of the Nonmetallic Materials Division, Materials Directorate, Wright Laboratory, with Mr. Kenneth M. Johnson (WL/MLBC) as Contract Monitor.

The use of commercial names of materials in this report is included for completeness and ease of scientific comparison only. This in no way constitutes an endorsement of these materials or manufacturers.

This report was submitted in February 1992 and covers work conducted from June 1990 to June 1991.

ACKNOWLEDGMENTS

This report would not have been possible without the support of numerous organizations and individuals both within the Air Force Materials Directorate and outside the Materials Directorate.

The author is especially grateful to Mrs. Becky Schiavone, former UDRI principal investigator, and to Dr. Allan Crasto for his many helpful suggestions throughout this task. Special thanks also go to Major Joseph Hager, WL/MLBC, for lending his organizational skills and numerous suggestions throughout the project.

The author would also like to acknowledge Mr. Mike Scott, Mr. Robert Lewis, and Mr. Eric Fletcher, on-site contractors for Universal Energy Systems, and the Characterization Facility (MLLM) for contributing both equipment and expertise which proved invaluable towards a significant portion of this report.

1. INTRODUCTION

Carbon-carbon composites are composed of a carbonaceous or graphitic matrix and a carbon or graphitic fiber reinforcement. These lightweight, advanced composites have the ability to withstand high-temperature environments up to 6000°F (3316°C), while maintaining or increasing its mechanical properties with temperatures up to approximately 3500°F (1927°C). The high-temperature capability makes carbon-carbon composites extremely attractive for a variety of aerospace structural applications such as thermal shields, control surfaces, engine components, and airframe structures.

A variety of carbon fibers can be used for reinforcement. The matrix resin is chosen based on its ability to yield a high carbon content after pyrolysis. Because carbon begins to oxidize around 800°F (427°C), a variety of oxygen barriers in the form of coatings or impregnants are used to prevent oxygen from reaching the carbon. Coating materials, to name a few, may include silicon carbide, boron carbide, and alumina. Inhibitors may be added to the matrix to protect the internal microstructure from oxidation. Inhibitors used include boron, zirconium diboride, and boron/silicon carbide particles. These many possible constituents, coupled with an underlying complex microstructure characterized by a network of voids, cracks, and fiber-matrix disbonds, create a challenge for the researcher to quantitatively and qualitatively characterize the microstructure. The challenge is further compounded if the carbon-carbon composite is to be characterized after various states of thermal oxidation.

The first approach was to capitalize on established characterization techniques presently available within the Materials Directorate to characterize and apply these to carbon-carbon composite characterization. This included techniques presently used to characterize both composites and metals. The Materials Directorate Characterization Facility, which utilizes state-of-the-art metallographic characterization techniques, provided equipment, supplies, and expertise which proved invaluable to this study. The Nondestructive Evaluation Facility within the Materials Directorate also provided support to evaluate computer tomography and ultrasonic techniques on carbon-carbon composites.

The second approach was to develop in-house carbon-carbon characterization techniques when existing techniques did not provide the desired information.

The third approach involved the submittal of carbon-carbon composite specimens to outside vendors and research facilities that utilize characterization techniques and equipment not available within the Wright Laboratory Materials Directorate.

This investigation concentrated on five areas of characterization: (1) sectioning, (2) specimen mounting, (3) polishing, (4) optical and electron microscopy, and (5) non-destructive evaluation.

2. EXPERIMENTAL

Two, two-dimensional carbon-carbon composite panels designated 8A and 8B were used for the bulk of this study. Both panels were co-processed by Hitco from prepreg of F064 phenolic resin and T-300 HT fiber, woven into a 5H satin cloth. The average panel thickness was 0.375 inch. The cure, carbonization, and graphitization cycles used to process these panels are outlined in Table 1 and Figure 1 [1]. Carbon-carbon composites using various reinforcement/matrix combinations and processing parameters were added to the study as they became available.

TABLE 1
PHENOLIC CURE CYCLE

Cycle	Temperature (°C)	Pressure (MPa)	Time (min)
Phenolic Resin Cycle			
Oven Debulk	74	---	30
Autoclave Debulk	74	1.86	30
Hydroclave Cycle	160	6.89	360

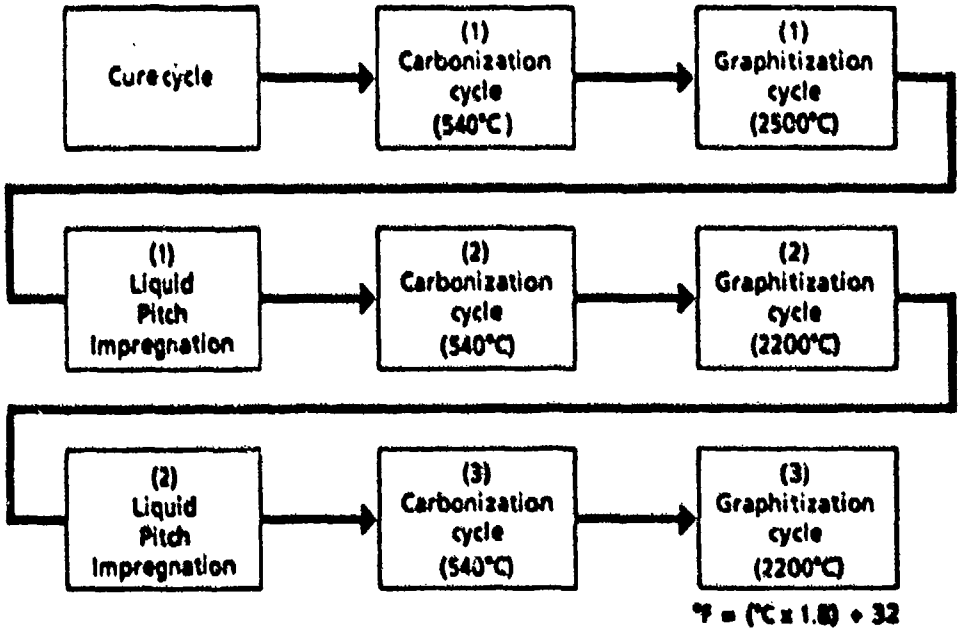


Figure 1. Hitco Processing Cycle for 8A and 8B Carbon-Carbon Composites.

3. SECTIONING TECHNIQUES

The purpose of this study was to determine the extent of surface and subsurface damage which may occur when machining or sectioning carbon-carbon composites. Ten different sectioning techniques were evaluated with the Hitco 8A composite panel; these included the use of four different circular saws, microtoming, milling, EDM wire arc, waterjet, ultrasonic, and laser sectioning techniques. The specimens were sectioned and evaluated as-received and after vacuum impregnation with an epoxy resin. The impregnation was accomplished in a Buehler vacuum impregnation chamber using Struers Epofix low-viscosity resin. The vacuum impregnation reinforces the porous, somewhat fragile internal structure to minimize damage during sectioning or polishing.

Details of the vacuum impregnation technique are described in Section 4.1. Scanning electron photomicrographs (SEMs) were taken of each cut surface to determine the type of damage incurred. A reliable technique to characterize subsurface damage was not available at this time.

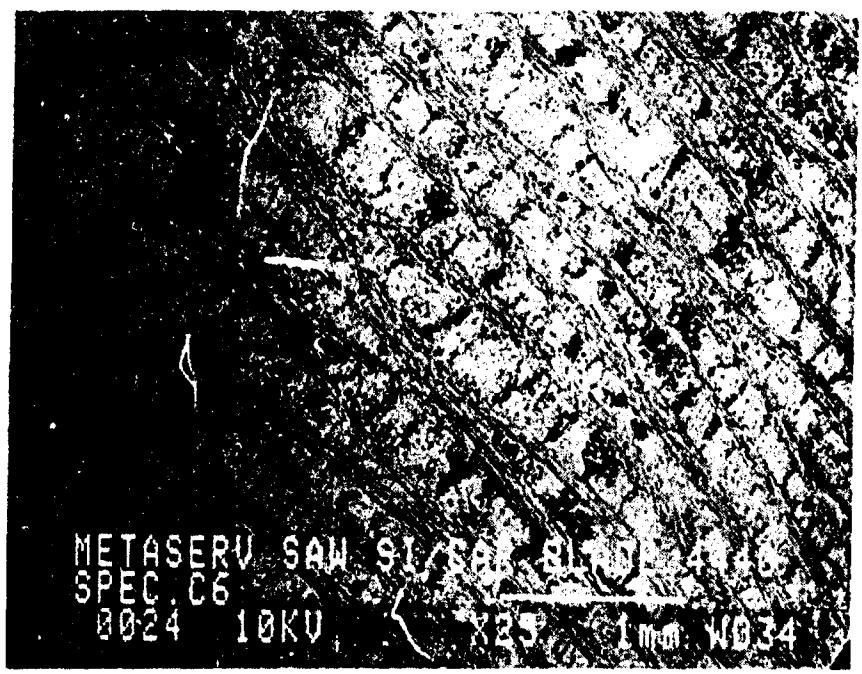
3.1 Circular Saw Sectioning

Four different circular saws were evaluated for carbon-carbon composite sectioning; a Metaserv saw from Buehler with a 4440 silicon carbide blade, a HT-14/20 Heathway saw with a metal-bonded diamond blade, a Buehler Isomet saw with a low-speed diamond blade, and a Struers Accutom saw with an aluminum oxide blade. The results are summarized below for nonvacuum-impregnated (NVI) and vacuum-impregnated (VI) samples.

3.1.1 Metaserv Saw

When comparing the VI sectioning SEM photomicrographs (Figure 2A) with the NVI SEM photomicrographs (Figure 2B), the VI sections lack voids that are clearly visible on the surface of the NVI sections. This indicates that the impregnation resin has infiltrated to the depth of the cut, filling or masking the voids. This was observed in all the section specimens that had been vacuum impregnated. The 0-degree fibers in both NVI and VI sections (Figure 2C) have a similar appearance. Fiber deformation and surface debris are common to both sections. The 90-degree fibers in the VI section (Figure 2D) appear to have less tearing or shredding when compared to the NVI sections.

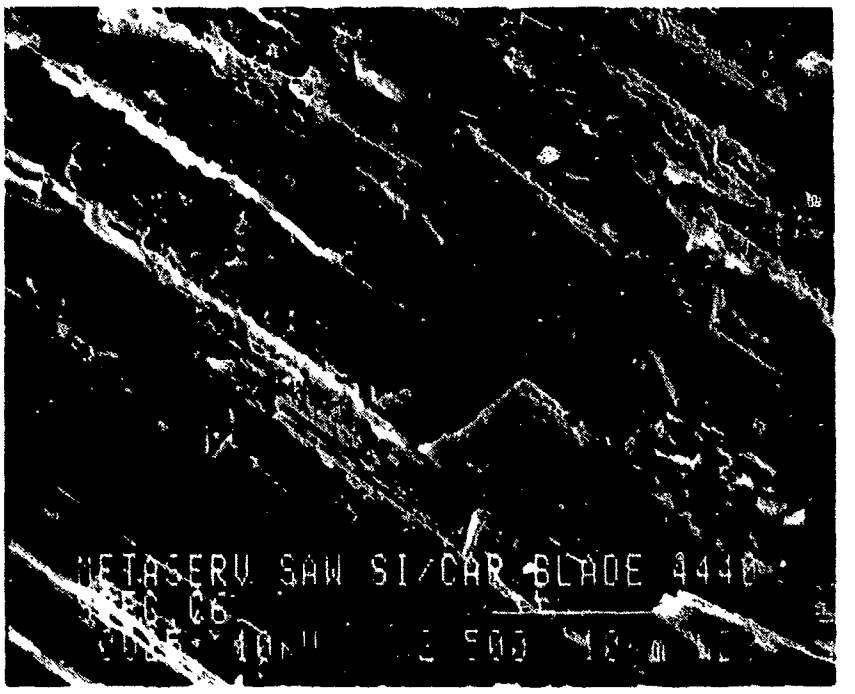
(a)



(b)

Figure 2. SEMs Showing Surface of Carbon-Carbon Specimen Sectioned on Metaserv Saw Using SiC Blade. (a) Vacuum Impregnated, 25X; (b) Nonvacuum Impregnated, 25X; (c) Vacuum Impregnated, 2500X; (d) Nonvacuum Impregnated, 2500X.

(c)



(d)

Figure 2 (Concluded). SEMs Showing Surface of Carbon-Carbon Specimen Sectioned on Metaserv Saw Using SiC Blade. (a) Vacuum Impregnated, 25X; (b) Non-vacuum Impregnated, 25X; (c) Vacuum Impregnated, 2500X; (d) Non-vacuum Impregnated, 2500X.

3.1.2 Heathway Saw

The transverse sections of the 0-degree fibers (Figure 3A) in both specimen types have similar rough-cut appearances. No significant differences between the two 90-degree fiber sections (Figure 3B) could be detected.

3.1.3 Isomet Saw

The 0-degree fiber surfaces in the VI sections (Figure 4A) appear to have a smoother, more even section with less surface debris when compared to the NVI sections (Figure 4B). The two 90-degree fiber sections (Figures 4C and 4D) are similar in appearance.

3.1.4 Accutom Saw

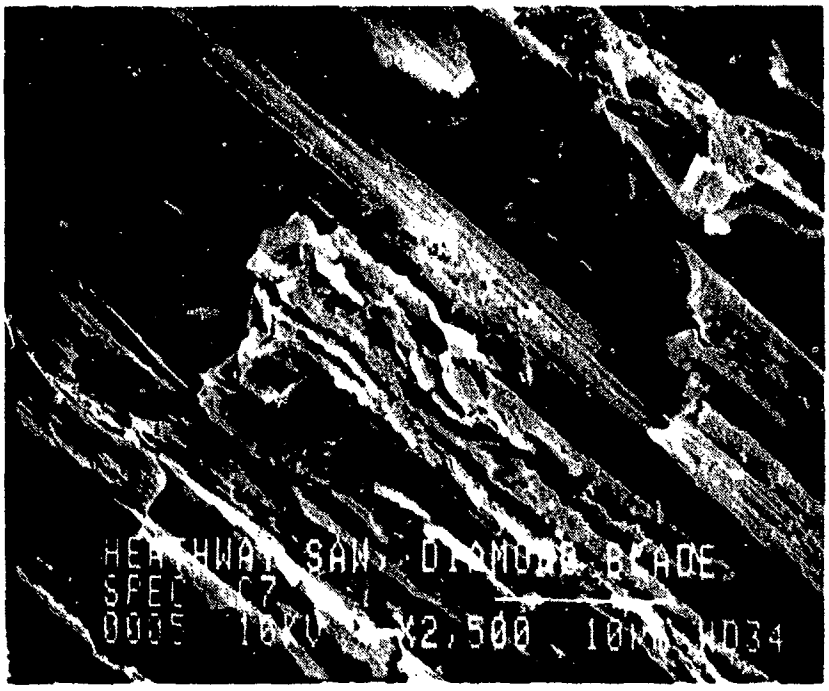
The 0-degree fibers in the VI sections (Figure 5A) appear to have a smoother cut with less surface debris than the NVI sections (Figure 5B). The 90-degree NVI fibers (Figure 5C) appear to have more damage than the 90-degree VI sections (Figure 5D). The difference in appearance between these two sections may have been due to replacement of the saw blade between the two cuts.

Among the saw sectioning techniques evaluated, the Accutom saw equipped with an aluminum-oxide blade produced less damage to the 0-degree fiber ends on both the VI and NVI sections (Figures 5A and 5B). Some of the 90-degree fibers were also sectioned more smoothly along the longitudinal axis, compared to the shredding observed with other saws. In conclusion, vacuum-impregnation with an epoxy resin reduces damage during the sectioning of these composites.

3.2 Ultrasonic Sectioning

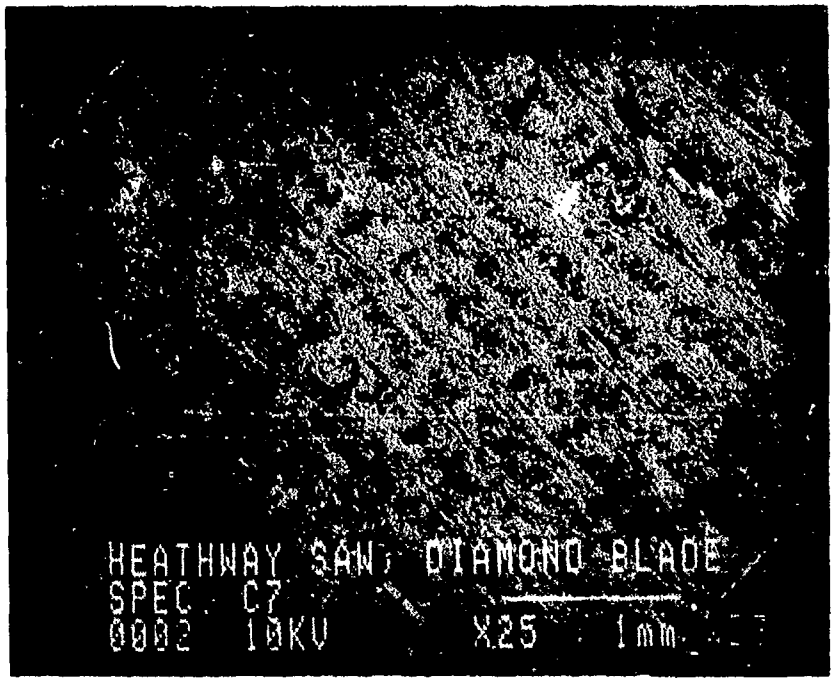
The Ultrasonic Cutter (Bendix Corporation) might be illustrated by reference to the pneumatic drill. The machine utilizes an oscillating chisel having sharp cutting edges at its tip. Material is removed through hammer blows by the chisel, the cutting edges chipping or spalling away particles of the workpiece. Additional cutting is achieved through abrasive grains flowing between the tool and the part. For this work a molyblade with a boron nitride abrasive was used. Progressively finer grit sizes up to paste forms can be used (depending on requirements), although only one abrasive was employed for this evaluation.

(a)

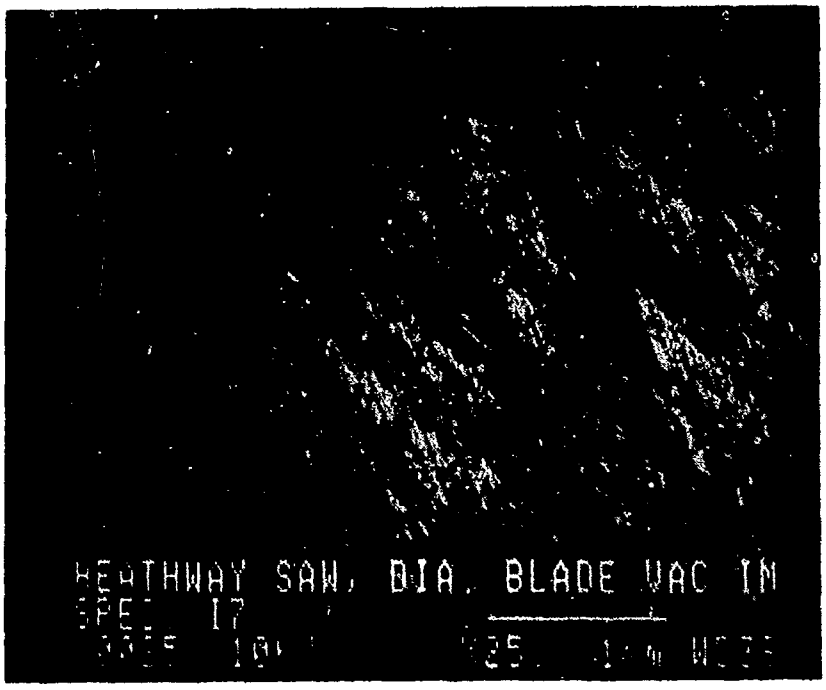


(b)

Figure 3. SEMs Showing Surface Area of Carbon-Carbon Specimen Sectioned on Heathway Saw Using Diamond Blade. (a) 0-Degree Fibers, Nonvacuum Impregnated, 2500X; (b) 90-Degree Fibers, Nonvacuum Impregnated, 2500X; (c) Nonvacuum Impregnated, 25X; (d) Vacuum Impregnated, 25X.



(c)



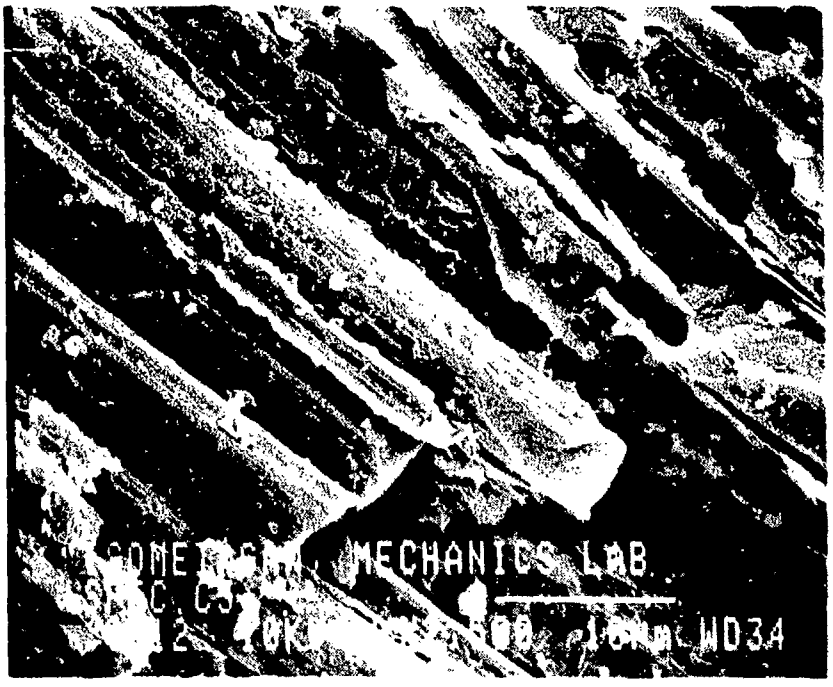
(d)

Figure 3 (Concluded). SEMs Showing Surface Area of Carbon-Carbon Specimen Sectioned on Heathway Saw Using Diamond Blade. (a) 0-Degree Fibers, Nonvacuum Impregnated, 2500X; (b) 90-Degree Fibers, Nonvacuum Impregnated, 2500X; (c) Nonvacuum Impregnated, 25X; (d) Vacuum Impregnated, 25X.

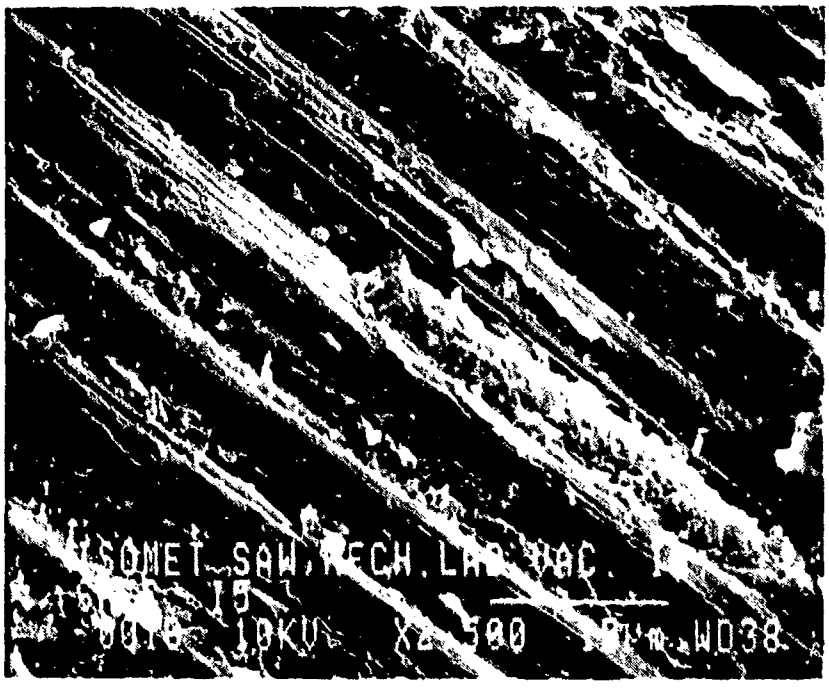
(a)

(b)

Figure 4. SEMs Showing Carbon-Carbon Specimen Sectioned on Isomet Saw Using Diamond Blade (2500X). (a) 0-Degree Fibers, Vacuum Impregnated; (b) 0-Degree Fibers, Nonvacuum Impregnated; (c) 90-Degree Fibers, Nonvacuum Impregnated; (d) 90-Degree Fibers, Vacuum Impregnated.

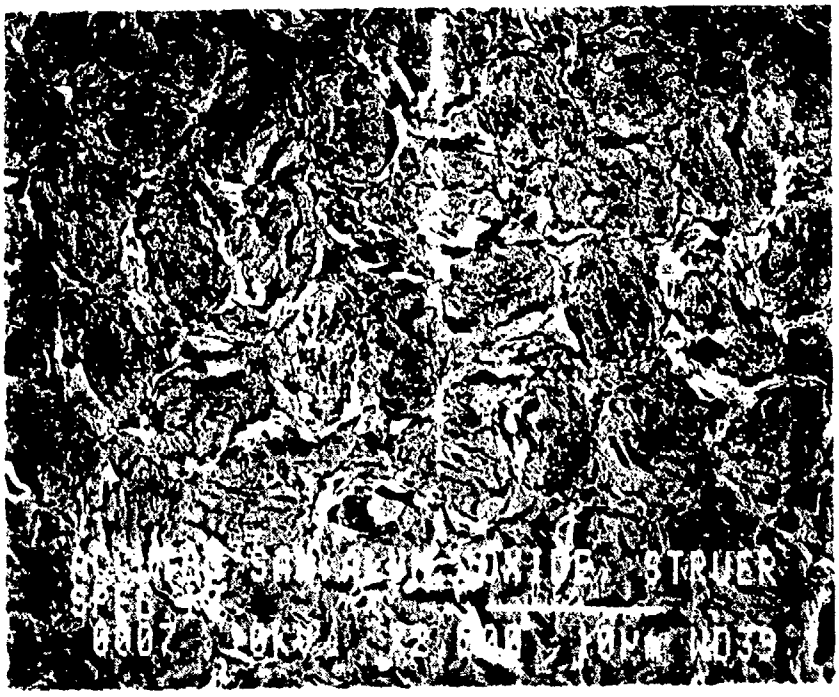


(c)

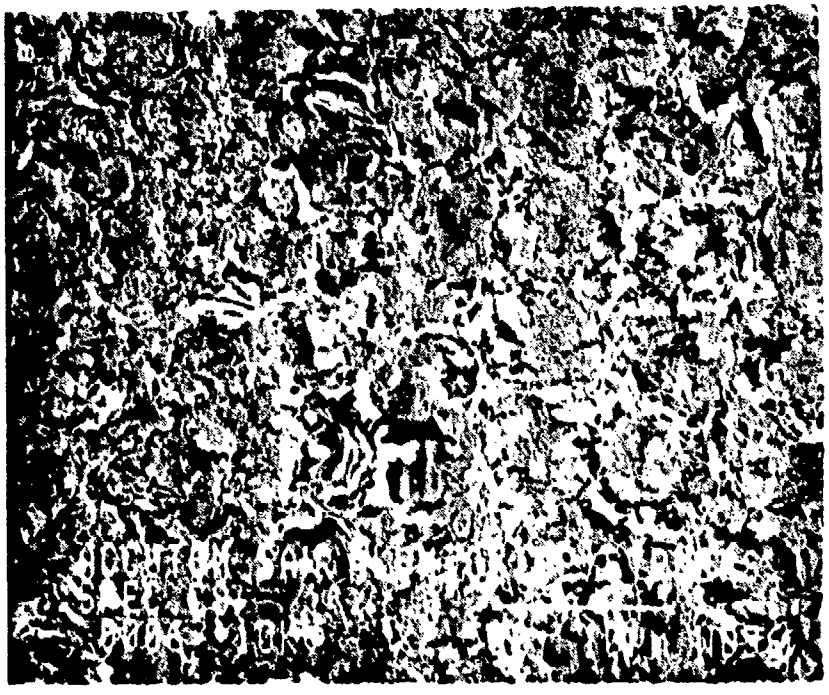


(d)

Figure 4 (Concluded). SEMs Showing Carbon-Carbon Specimen Sectioned on Isomet Saw Using Diamond Blade (2500X). (a) 0-Degree Fibers, Vacuum Impregnated; (b) 0-Degree Fibers, Nonvacuum Impregnated; (c) 90-Degree Fibers, Non-vacuum Impregnated; (d) 90-Degree Fibers, Vacuum Impregnated.



(a)



(b)

Figure 5. SEMs Showing Carbon-Carbon Specimen Sectioned on Accutom Saw Using Aluminum Oxide Blade. (a) 0-Degree Fibers, Vacuum Impregnated, 2500X; (b) 0-Degree Fibers, Nonvacuum Impregnated, 2500X; (c) Nonvacuum Impregnated, 150X; (d) Vacuum Impregnated, 150X.

(c)

(d)

Figure 5 (Concluded). SEMs Showing Carbon-Carbon Specimen Sectioned on Accutom Saw Using Aluminum Oxide Blade. (a) 0-Degree Fibers, Vacuum Impregnated, 2500X; (b) 0-Degree Fibers, Nonvacuum Impregnated, 2500X; (c) Nonvacuum Impregnated, 150X; (d) Vacuum Impregnated, 150X.

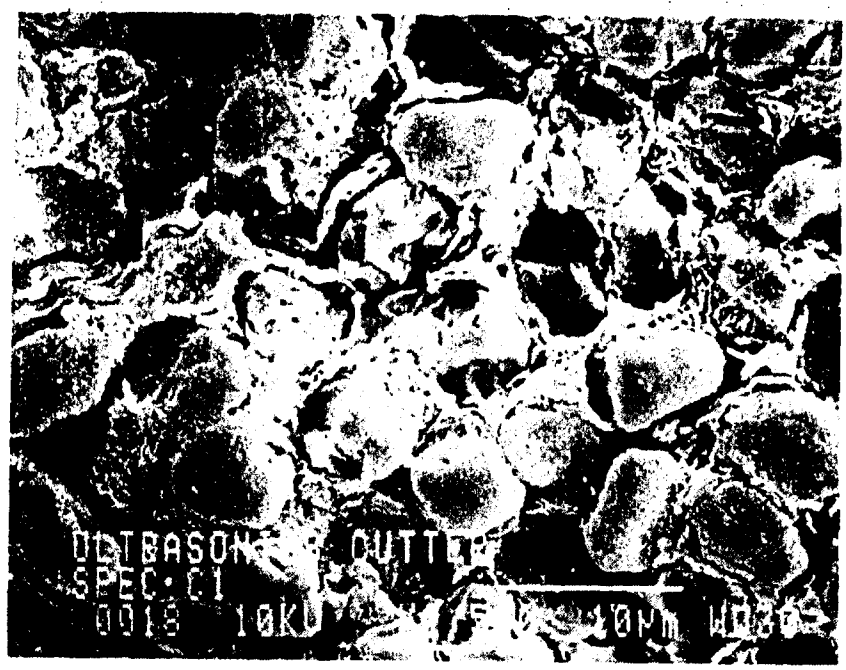
The ends of the 0-degree fibers in the NVI sections (Figure 6A) have a smeared, rounded appearance. There is indication of severe damage to the brittle matrix and at the fiber-matrix interface (Figures 6A and 6D). Voids in both specimens appear to be enlarged by the sectioning process (Figure 6B). In the VI sections the fiber ends appear to be broken or snapped (Figure 6C), rather than cut, and some cross-section surface detail is evident. This sectioning method is time consuming and restricted to simple straight cuts.

3.3 Water-Jet Sectioning

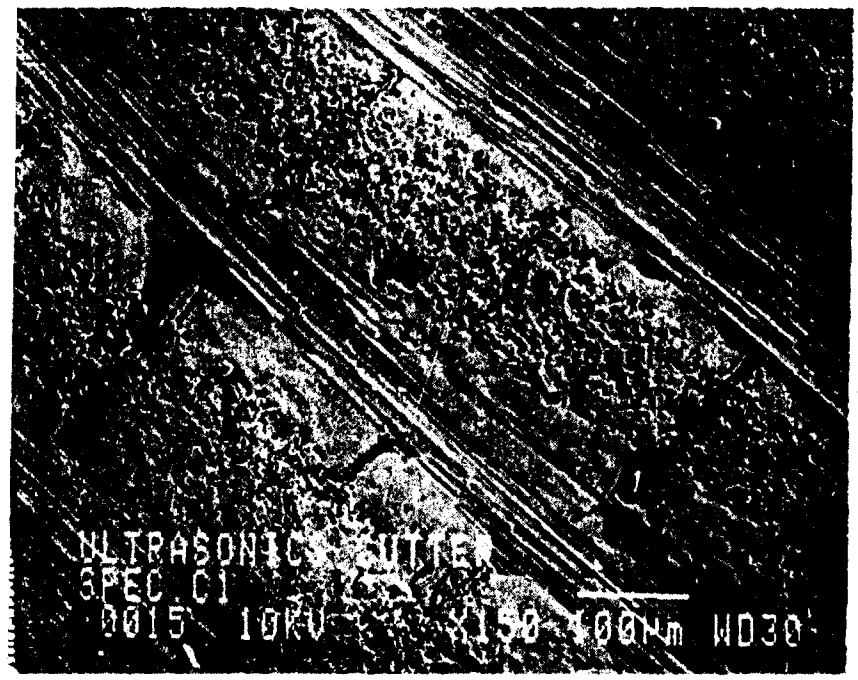
Water-jet sectioning is a relatively new technique. Abrasive water-jet cutting is especially suited to nonhomogeneous materials that are abrasive in nature and damaging to conventional cutting tools, or materials that produce dust or toxic fumes during cutting. This technique can also be used to cut intricate shapes.

Water-jets are driven hydraulically. An intensifier unit pumps a filtered fluid at pressures up to 410 MPa (60 ksi) through an orifice to form a jet stream. An abrasive material can be added to the fluid to aid in cutting. Typically a garnet abrasive with grit size ranging from 16 to 150 mesh is used. There are several benefits to be realized by using this sectioning technique. When the water-jet is allowed to dwell on a piece, the kerf is generally not affected. The kerf is relatively narrow (its width is dependent on the exit nozzle bore size), and a minimum of material is therefore removed. Due to the low forces applied by the water-jet, the workpiece, in most cases, can be held in place with simple weights. There is no noticeable heat generated due to the low cutting temperature and very little dust produced by the process.

A problem that might occur is composite delamination, if the cutting action is started from the edge of the piece. It is suggested that the jet be activated away from the edge of the workpiece and then guided into the workpiece. In addition, insufficient velocity or a loss of abrasive can also initiate delamination. High noise levels are generated by air coupling into a high-velocity large volume of air/water, and ear protection must be worn during operation. Another concern with this method is that the jet stream tends to angle away from the cutting direction. This effect becomes more severe with an increase in either the thickness of the workpiece or the nozzle feed rate. Abrasive cutting also produces a wider kerf at the entrance of the cut, and this effect will vary with the jet speed. Whether or not this effect is a problem depends on the particular application. Another consideration in abrasive water-jet cutting is the surface finish of the cut edge; the finer the abrasive mesh, the smoother the finished edge.

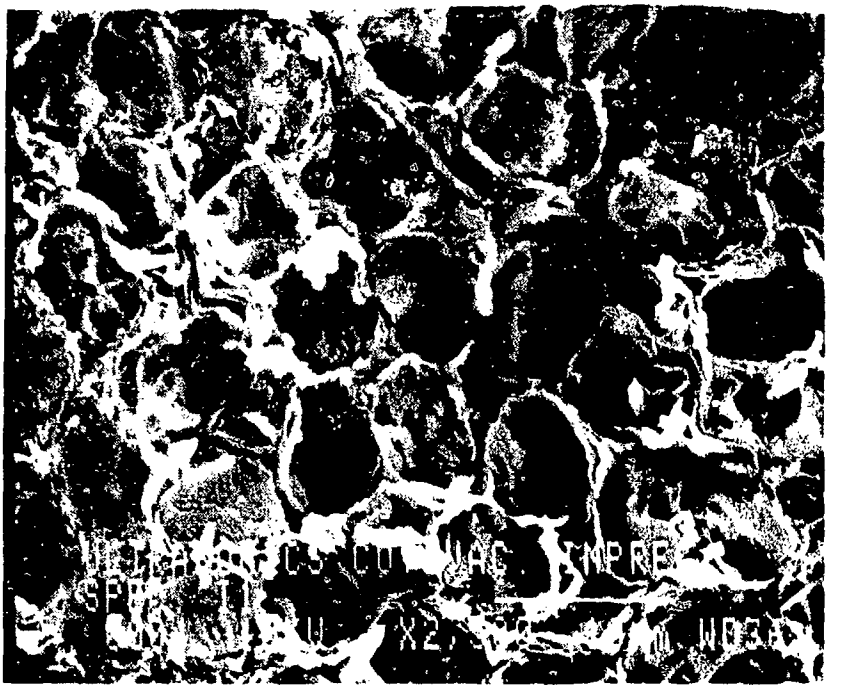


(a)

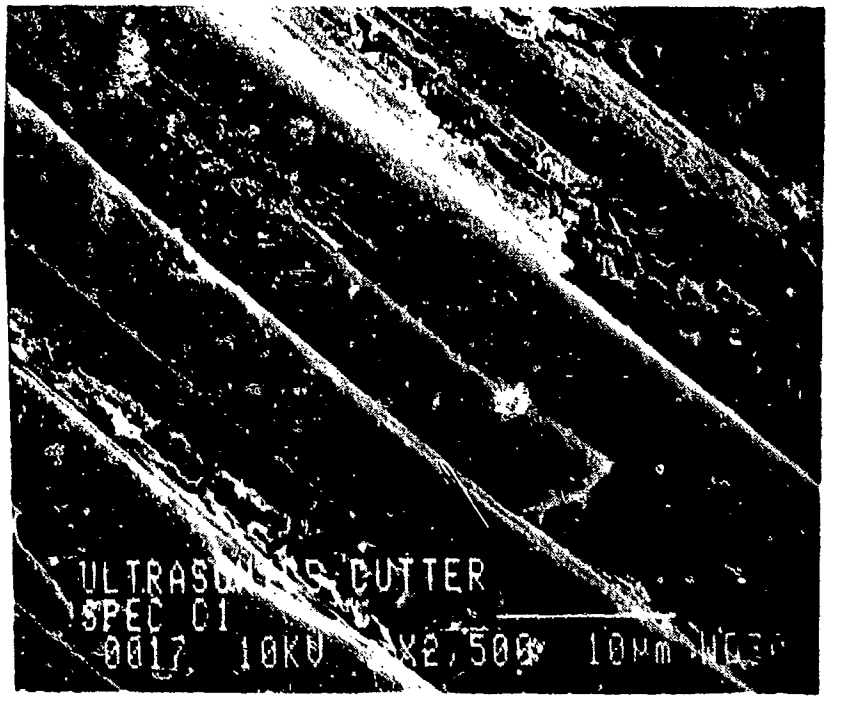


(b)

Figure 6. SEMs Showing Carbon-Carbon Specimen Sectioned on Ultrasonic Cutter. (a) 0-Degree Fibers, Nonvacuum Impregnated, 2500X; (b) Nonvacuum Impregnated, 150X; (c) 0-Degree Fibers, Vacuum Impregnated, 2500X; (d) 90-Degree Fibers, Nonvacuum Impregnated, 2500X.



(c)



(d)

Figure 6 (Concluded). SEMs Showing Carbon-Carbon Specimen Sectioned on Ultrasonic Cutter. (a) 0-Degree Fibers, Nonvacuum Impregnated, 2500X; (b) Nonvacuum Impregnated, 150X; (c) 0-Degree Fibers, Vacuum Impregnated, 2500X; (d) 90-Degree Fibers, Nonvacuum Impregnated, 2500X.

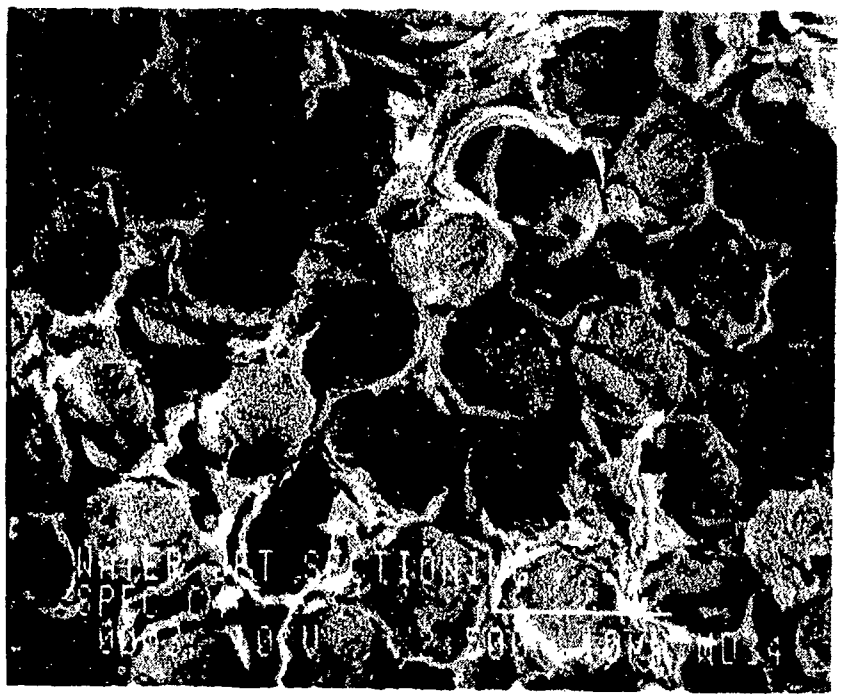
An Ingersoll-Rand water-jet cutter, Model HK-8-05, was used for this evaluation. A 120-grit garnet abrasive was used to facilitate cutting and to create as smooth an edge as possible. All 0-degree fibers appear to be snapped or sheared off at different depths (Figure 7A). The 90-degree VI sections (Figure 7B) appeared to have somewhat smoother cuts than the 90-degree NVI sections (Figure 7C).

Carbon-carbon composites have been machined on multiaxis water-jet cutters at different facilities with excellent results reported. Gaining experience and establishing optimum operating parameters for carbon-carbon composites will be necessary to improve water-jet sectioning results at this facility.

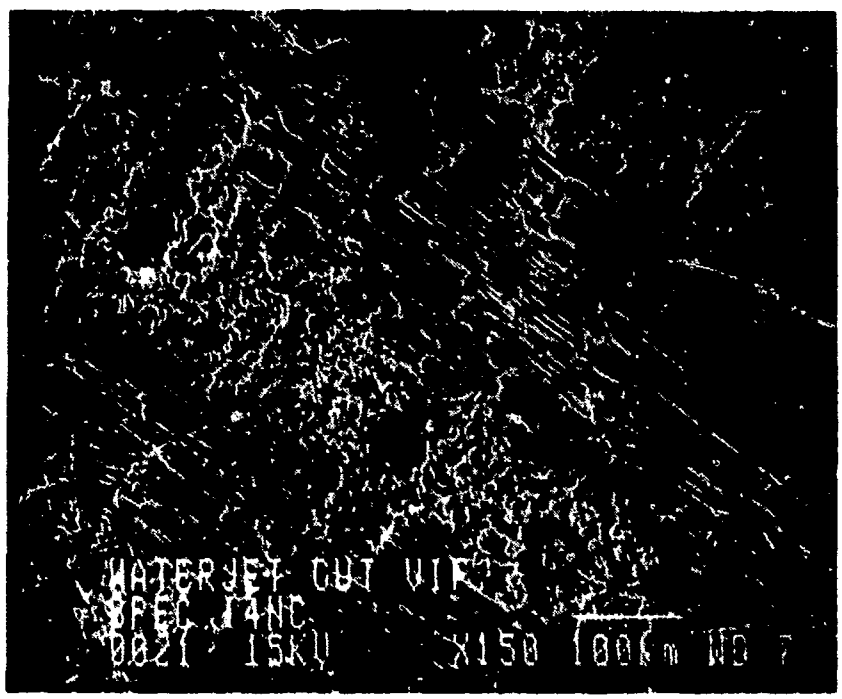
3.4 Electro Discharge Machining (EDM)

Sectioning by electro discharge was accomplished on one of two machines located at the 4950th Test Wing/Zone Machine Shop, WPAFB. As its name implies, EDM uses an electrical discharge or arc to erode the material being sectioned. The source of the electrical discharge is a brass wire that is continuously fed past the workpiece. The wire is not reused. The workpiece can be clamped or secured to the table with double-sided adhesive tape and the table programmed to travel on five different axes, giving it a large degree of flexibility. The kerf or cut width depends on the wire diameter. This study employed a 0.012-inch wire which, along with an overburn of 0.002 inch, resulted in a kerf of 0.014 inch. The type of wire used depends on such factors as the material being sectioned, the thickness of the workpiece, and the desired cutting speed. Both VI and NVI specimens were evaluated with this technique.

The 0-degree fibers on the NVI sections (Figure 8A) appear to be smoothly cut. Some interfacial debonding is evident. From an examination of the entire surface, the porosity of both NVI and VI sections appear to be considerably enlarged as in Figure 8B. The ends of the 90-degree fibers in the VI sections (Figure 8C) are spear-shaped indicating severe oxidation, while the 0-degree fiber ends (Figure 8D) were severely eroded. These observed differences between VI and NVI sections may possibly be due to differences in machining parameters. The VI specimen was difficult to cut. The electrical discharge wire broke on several occasions. Consequently both the speed and voltage were changed to prevent this from occurring. These parameter changes may have induced oxidation of the sectioned VI surfaces.



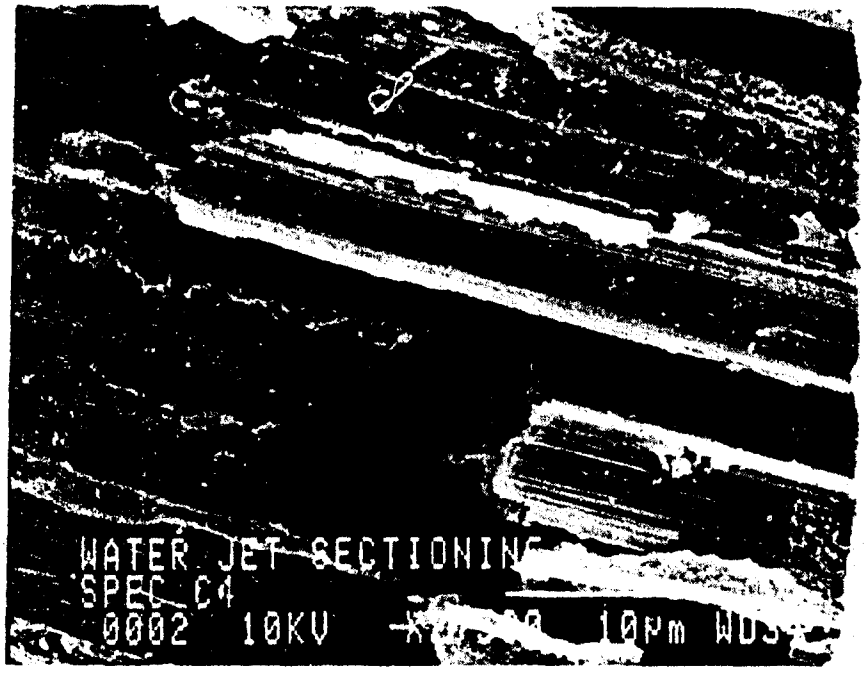
(a)



(b)

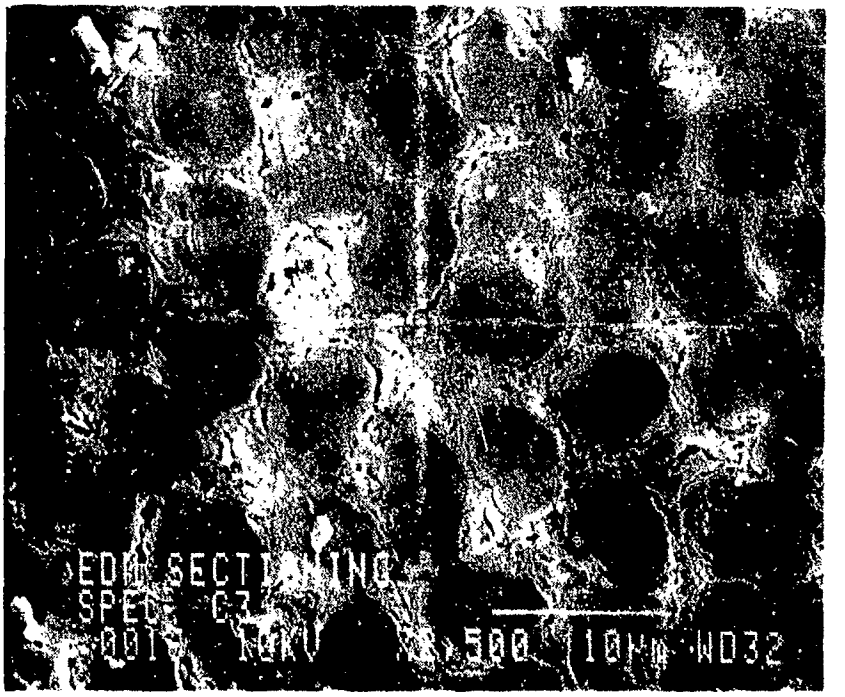
Figure 7. SEMs Showing Carbon-Carbon Specimen Sectioned on Water-Jet Cutter. (a) 0-Degree Fibers, Nonvacuum Impregnated, 2500X; (b) Vacuum Impregnated, 150X; (c) Nonvacuum Impregnated, 150X; (d) 90-Degree Fibers, Nonvacuum Impregnated, 2500X.

(c)

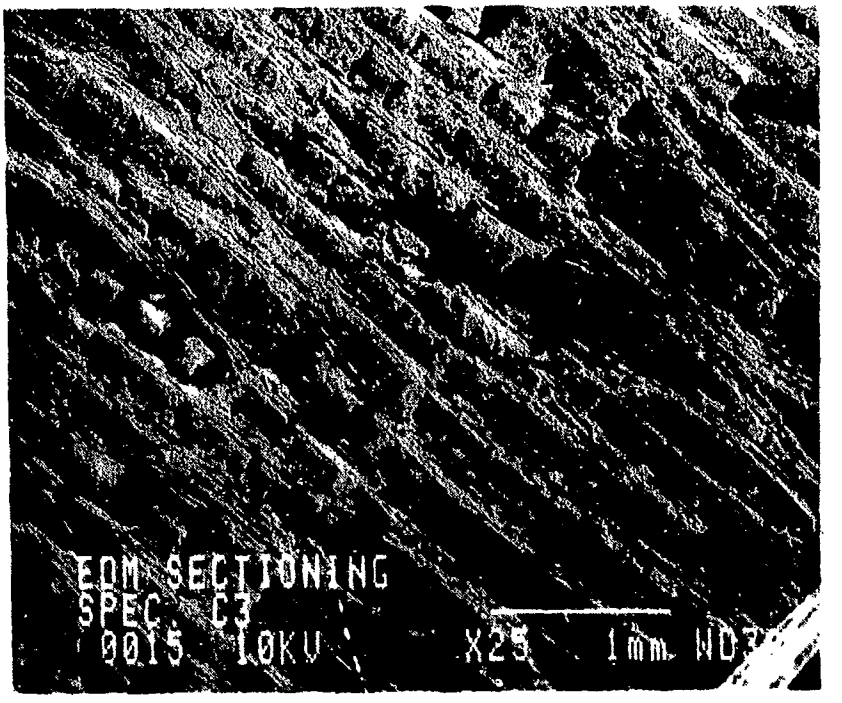


(d)

Figure 7 (Concluded). SEMs Showing Carbon-Carbon Specimen Sectioned on Water-Jet Cutter. (a) 0-Degree Fibers, Nonvacuum Impregnated, 2500X; (b) Vacuum Impregnated, 150X; (c) Nonvacuum Impregnated. 150X; (d) 90-Degree Fibers, Nonvacuum Impregnated, 2500X.

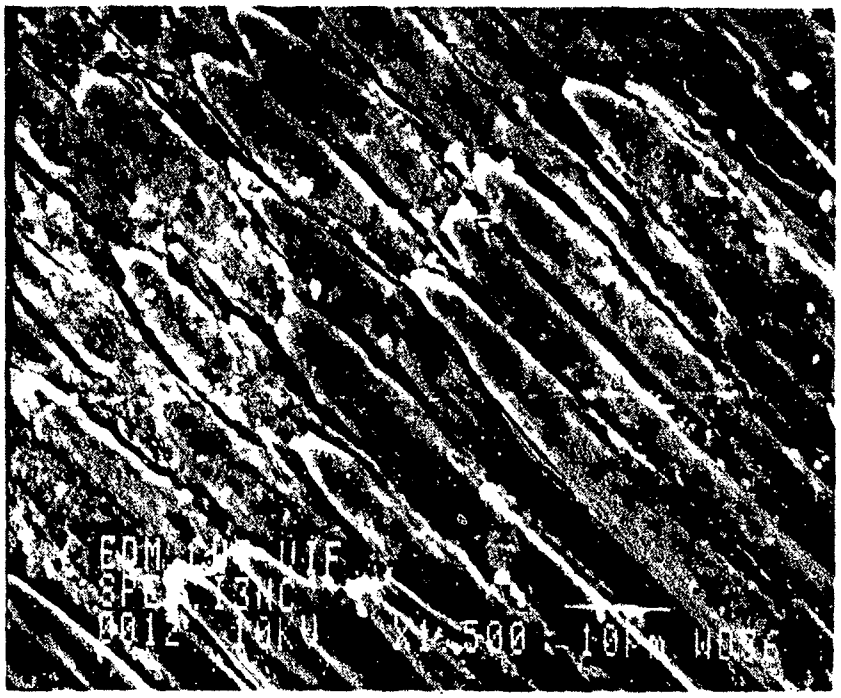


(a)

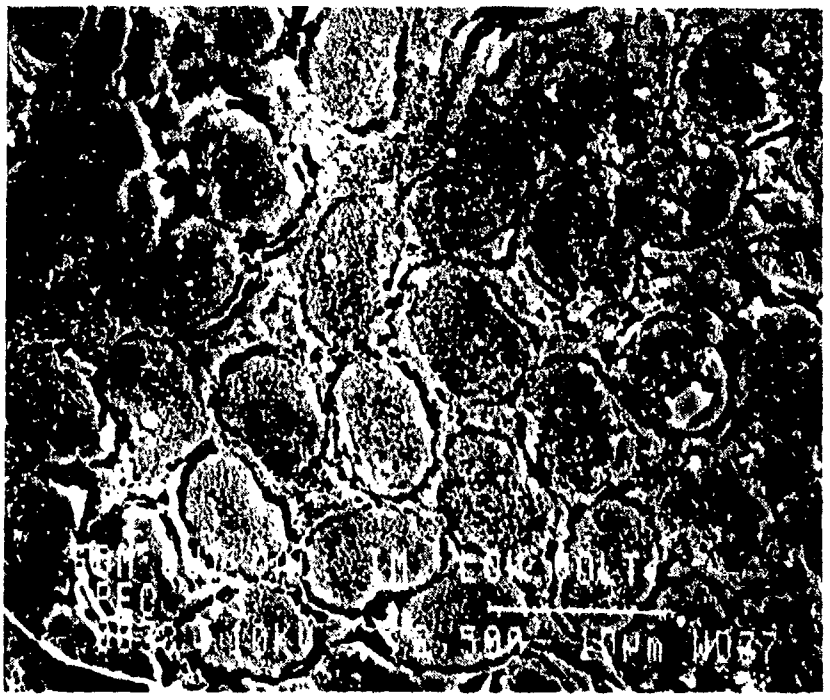


(b)

Figure 8. SEMs Showing Carbon-Carbon Specimen Sectioned on EDM Apparatus. (a) 0-Degree Fibers, Nonvacuum Impregnated, 2500X; (b) Nonvacuum Impregnated, 25X; (c) 90-Degree Fibers, Vacuum Impregnated, 1500X; (d) 0-Degree Fibers, Vacuum Impregnated, 2500X.



(c)



(d)

Figure 8 (Concluded). SEMs Showing Carbon-Carbon Specimen Sectioned on EDM Apparatus. (a) 0-Degree Fibers, Nonvacuum Impregnated, 2500X; (b) Non-vacuum Impregnated, 25X; (c) 90-Degree Fibers, Vacuum Impregnated, 1500X; (d) 0-Degree Fibers, Vacuum Impregnated, 2500X.

3.5 Laser Sectioning

Laser sectioning has several advantages over conventional machining. Since the contact to the surface of the workpiece is a thermal process, this allows for intricate cutting of fragile materials. Composites which prove difficult to machine with conventional methods are more often than not easily cut with a laser. As an example aramid-epoxy composites which are otherwise intractable are easily cut by a CO_2 laser. While laser sectioning offers an alternative to other machining methods, the results for carbon-carbon composites were not encouraging. Thermal degradation or charring was evident on all specimen surfaces sectioned using this method (Figures 9A through 9D). The laser essentially relies on vaporization to achieve its task. Again, additional experience cutting this material with the laser may yield better results.

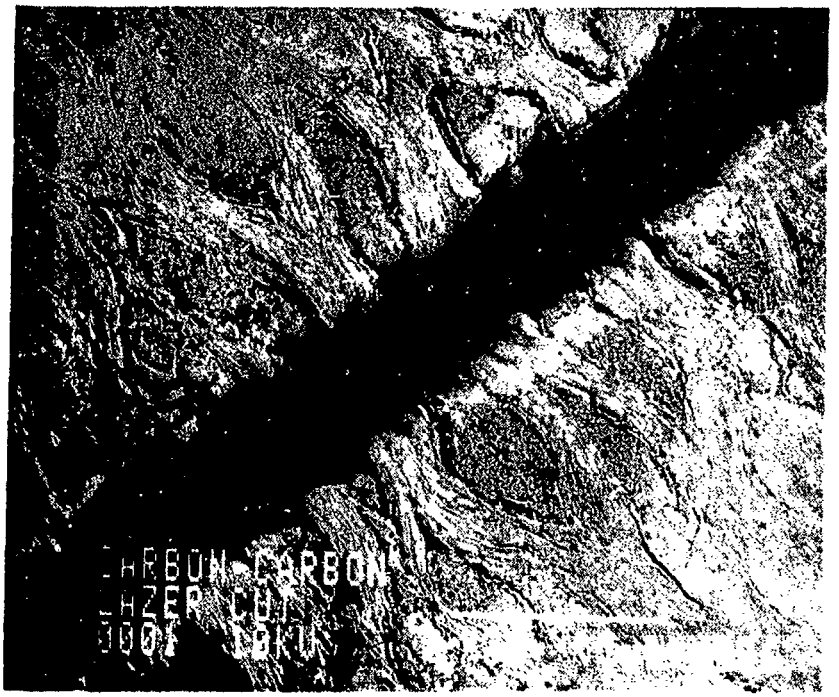
3.6 Microtome Sectioning

Microtomes are widely used to create thin slices with controlled thicknesses for microscopic examination. In this study a Polycut E microtome from Reichert-Jung was used to section carbon-carbon composites. Various blades were employed without success, as the specimens disintegrated in each case. The Polycut E can be fitted with an attachment called an ultramiller. This technique is described below.

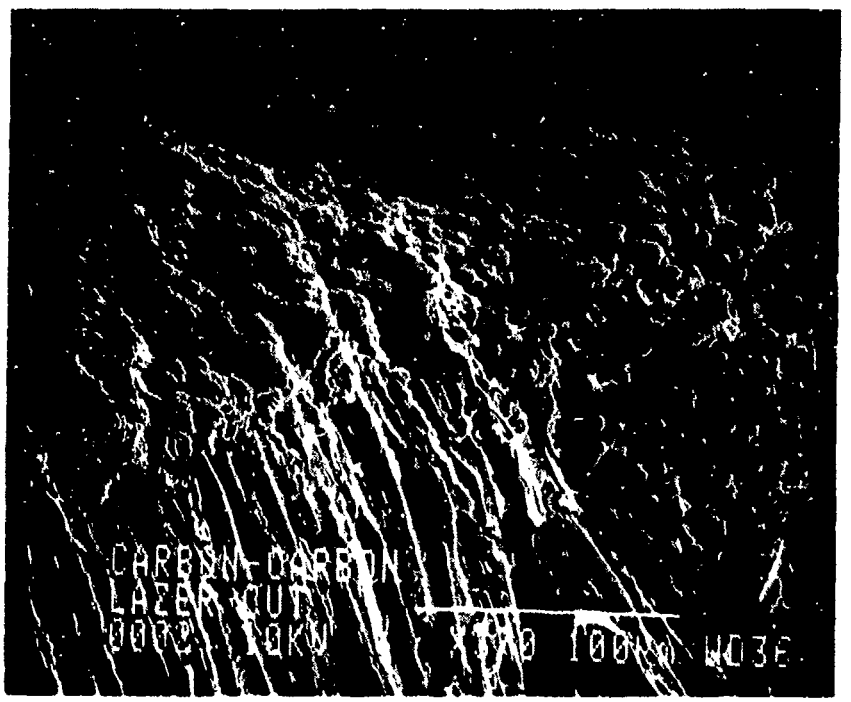
3.7 Ultramilling

The ultramiller, as the name suggests, is a milling technique rather than a sectioning technique. The apparatus is designed to precisely thin down a roughly-cut specimen for inspection in an optical or electron microscope. This method is capable of producing flat surfaces with an undulation of less than 0.1 micron, excellent edge definition, and no smearing. In this method a rotating vertical spindle with a pair of diamond cutters removes a thin layer of material from the specimen which is driven under the cutters. The two cutters rotate on arms of different radii. As the sample travels past the rotating heads, the head with the greater radius premills the sample. The diagonally opposite head at a lower radius then finely mills the surface.

The 0-degree fibers in the NVI section (Figure 10A) appear to be snapped off, with considerable surface debris left on the surface. The 0-degree fiber surfaces of the VI section are somewhat smoother in appearance. The 90-degree fibers of the NVI specimen (Figure 10B) appear to have less fiber breakage than the other sectioning techniques attempted. Observing the VI specimen, the 90-degree fibers (Figure 10C) have considerably more breakage and surface debris than the 90-degree NVI section (Figure 10D).



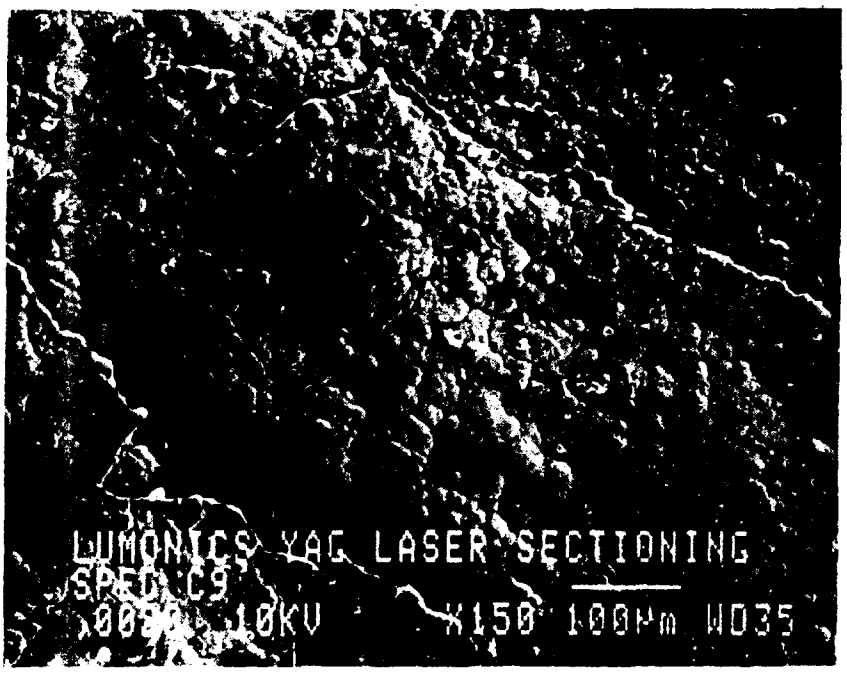
(a)



(b)

Figure 9. SEMs Showing Severe Oxidation on Surface of Laser Sectioned Carbon-Carbon Specimen. (a) 30X; (b) 370X; (c) 25X; (d) 150X.

(c)



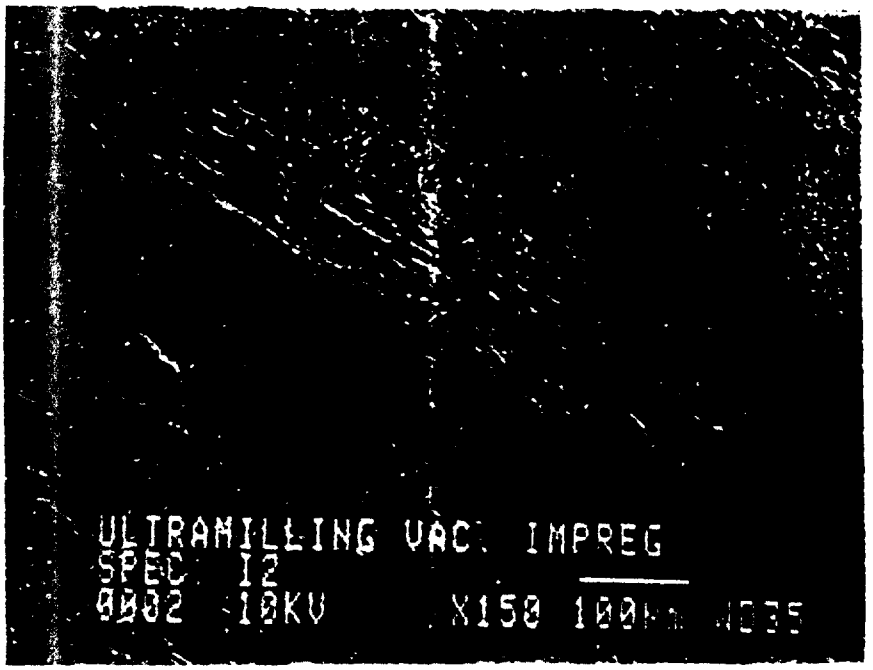
(d)

Figure 9 (Concluded). SEMs Showing Severe Oxidation on Surface of Laser Sectioned Carbon-Carbon Specimen. (a) 30X; (b) 370X; (c) 25X; (d) 150X.

(a)

(b)

Figure 10. SEMs Showing Carbon-Carbon Specimen Sectioned on Ultramiller. (a) 0-Degree Fibers, Nonvacuum Impregnated, 2500X; (b) 90-Degree Fibers, Nonvacuum Impregnated, 2500X; (c) Vacuum Impregnated, 150X; (d) Non-vacuum Impregnated, 150X.



(c)

(d)

Figure 10 (Concluded). SEMs Showing Carbon-Carbon Specimen Sectioned on Ultramiller. (a) 0-Degree Fibers, Nonvacuum Impregnated, 2500X; (b) 90-Degree Fibers, Nonvacuum Impregnated, 2500X; (c) Vacuum Impregnated, 150X; (d) Non-vacuum Impregnated, 150X.

The sectioning technique used for carbon-carbon composites depends on the features of interest. Some techniques more efficiently section the matrix, while other techniques section the fibers more efficiently. The sectioned surfaces of fibers are more influenced by the technique employed than are matrix areas. Vacuum impregnation minimizes surface and possibly subsurface damage to some degree.

4. VACUUM/PRESSURE IMPREGNATION

Vacuum impregnation with an epoxy resin was discussed briefly in the previous section and will now be discussed in detail. In addition vacuum impregnation with resins tagged with a fluorescent dye and with low melting alloys will be discussed.

4.1 Vacuum Impregnation with Epoxy Resin

Vacuum impregnation is used to fill the cavities of porous materials when those cavities are open to the surface. This is done primarily to preserve intact the internal microstructure during subsequent specimen preparation procedures. The support provided by the impregnant minimizes fiber tearout, delamination, and enlargement of cracks and voids during sectioning and polishing. Retention of the original cross section can ensure an accurate measurement of porosity.

A vacuum impregnation apparatus available through Buehler Products (Figure 11) was used for the bulk of this work. The main body of this apparatus is essentially a modified desiccator. There is an evacuation port on the lid and a gage to measure the rough vacuum. A pouring device is mounted inside the chamber in which a cup containing the impregnating resin may be placed. A handle outside the chamber allows the resin to be poured while the chamber is evacuated. An electrically-controlled rotating stage allows the impregnation of multiple specimens from one container of resin. A low-viscosity epoxy resin, Epofix, from Struers, Inc. was used as the impregnant. This two-component resin system has good wetting characteristics and negligible heat generation and shrinkage on hardening.

The vacuum impregnation process was conducted as follows. A cup containing the premixed, low-viscosity resin system was placed in the pouring device within the chamber. The specimen was placed in a plastic container (Sampl-Kup from Buehler), the inside of which was coated with a mold release agent. This container was placed within the chamber which is then evacuated with a standard laboratory pump. Vacuum was held for one minute to completely evacuate the open pores within the specimen. The resin was then poured over the specimen, completely submerging it. The resin is allowed to cure at room temperature for 12 hours. Once cured, the specimen can be sectioned or polished while still embedded in the epoxy plug, or the plug may be carefully sanded or cut away to free the specimen.

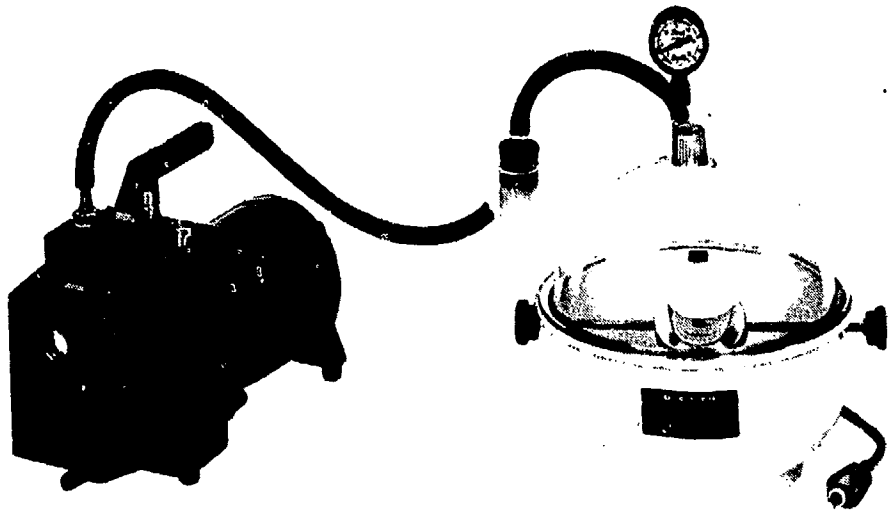


Figure 11. Buehler Vacuum Impregnation Chamber.

4.2 Impregnation with a Fluorescent Dye

Various materials emit light of a longer wavelength when they are excited by short wavelengths of radiation. This phenomenon is called fluorescence. Ultraviolet radiation and blue light are often used as the exciting radiation to produce visible light at longer wavelengths. This exciting radiation is produced by a high-pressure mercury vapor or xenon lamp and an exciter filter employed to absorb the visible light generated. The cracks and pores in carbon-carbon composites can be sharply contrasted to the solid material in an optical microscope if the impregnating resin is tagged with a fluorescent dye. Dyes used in this manner are called fluoro-chromes [2]. A Nikon Microphot FXL microscope equipped for fluorescence photomicroscopy and a Reichert-Jung MeF3-A inverted microscope equipped with a 400-W xenon arc lamp were used to optically enhance the fluorescent dye that infiltrated the open cavities of the composite.

In this study 0.75-inch x 0.75-inch x 0.375-inch 8A carbon-carbon specimens were vacuum impregnated with Epofix resin tagged with Epodye, a fluorescent dye from Struers, Inc. The dye, which is in powder form, was mechanically stirred into the resin (five grams dye to one liter of resin) until a homogeneous mixture was obtained. Resin-impregnated specimens were cross sectioned three times towards the middle, polished, and examined. Photomicrographs at magnifications between 50X and 400X indicated only partial infiltration of open porosity with the tagged resin with increasing depth. Brightfield illumination at 6.3X (the entire cross section

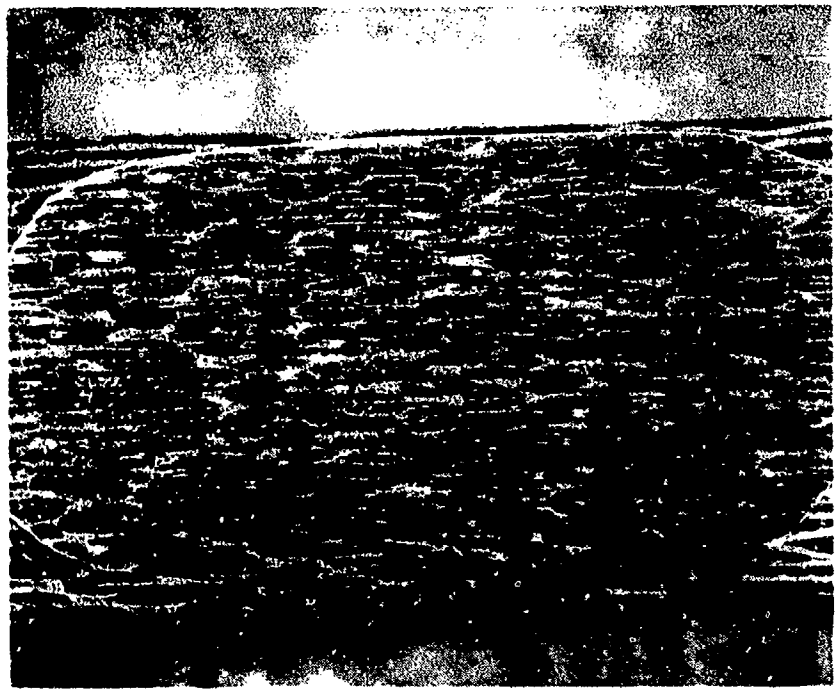
is visible) clearly demonstrates this phenomenon. Figure 12A is an example of a polished carbon-carbon composite that has not been resin impregnated. Figure 12B is an example of the same material that has been partially infiltrated by the impregnation resin (oval-shaped area in center represents nonimpregnated area). The voids in this central region appear enlarged by the sectioning/polishing steps. This lack of complete infiltration may be due to incomplete evacuation of the specimen cavities prior to addition of the resin. The evacuation time is kept as short as possible to prevent outgassing of the hardener and/or foaming of the resin mixture which is also simultaneously being evacuated. Figure 13 is an example of oxidized Hitco 8A carbon-carbon that has been vacuum impregnated with a fluorescent resin. The enlarged cavities created by the elevated temperature were easily impregnated and are greatly enhanced using this technique. A vacuum impregnation chamber is now available from Struers (Figure 14) which allows introduction of the resin through a tube running to a resin reservoir located outside the chamber. This allows evacuation of the specimen within the chamber for as long as necessary to displace all air from the open porosity and cracks of the specimen.

Alternatively, pressure may be applied to the specimen subsequent to the resin addition to aid in achieving complete impregnation. Pressure impregnation is described in the following section.

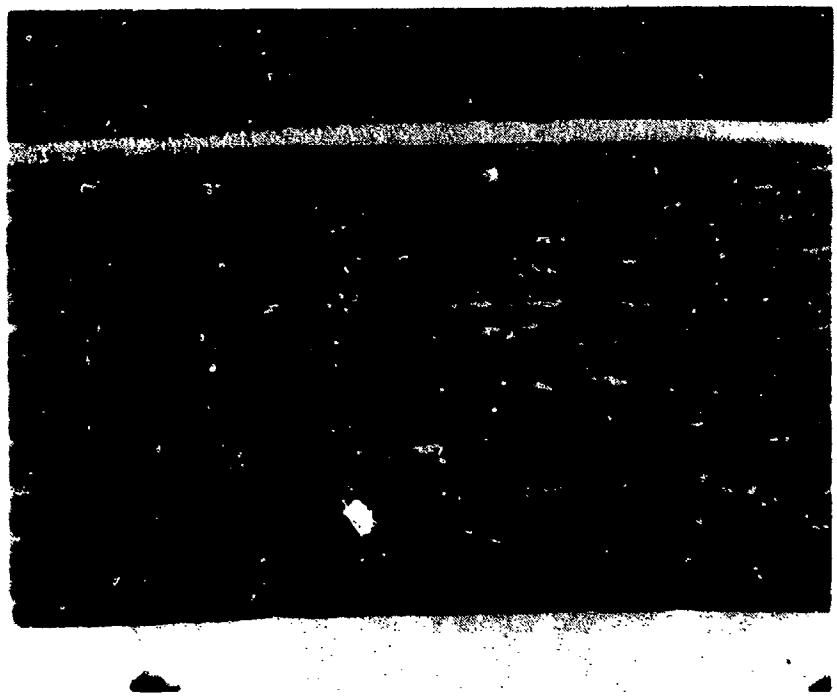
4.3 Impregnation Under Pressure

Impregnation under isostatic pressure was performed in a hot isostatic press or HIP (Figure 15) which is designed to consolidate metals at elevated temperatures and pressures. The carbon-carbon specimen was cleaned ultrasonically in acetone for five minutes and then heated in a vacuum oven to remove residual solvent. The specimen was then secured at the bottom of a 30-ml beaker with a steel wire arrangement (Figure 16) to prevent the specimen from floating to the surface before impregnation. For this study another epoxy resin system was employed, Epon 828 (from Shell Chemical Company) cured with Jeffamine D230 (a polyetheramine hardener from Texaco Chemical Corporation). This system was employed due to its lower viscosity and lower vapor pressure. The specimen was vacuum impregnated as described earlier and then pressurized in the HIP at 1 ksi for 30 minutes. Following this step the resin containing the submerged specimen was cured in a forced convection oven at 65°C for two hours.

Several specimen sections were examined and once again revealed only partial impregnation (Figures 17A and 17B). Through further experimentation it was determined that an impregnation pressure of at least 5 ksi was required to ensure full resin infiltration (Figures 18A and 18B). This pressure is not so high as to damage the internal structure. Closed porosity (not



(a)



(b)

Figure 12. Optical Photographs Showing Surface of Carbon-Carbon Specimen. (a) Non-vacuum Impregnated, Enlarged Porosity Created by Sectioning and Polishing Process; (b) Vacuum Impregnated, Noninfiltrated Area in Center.

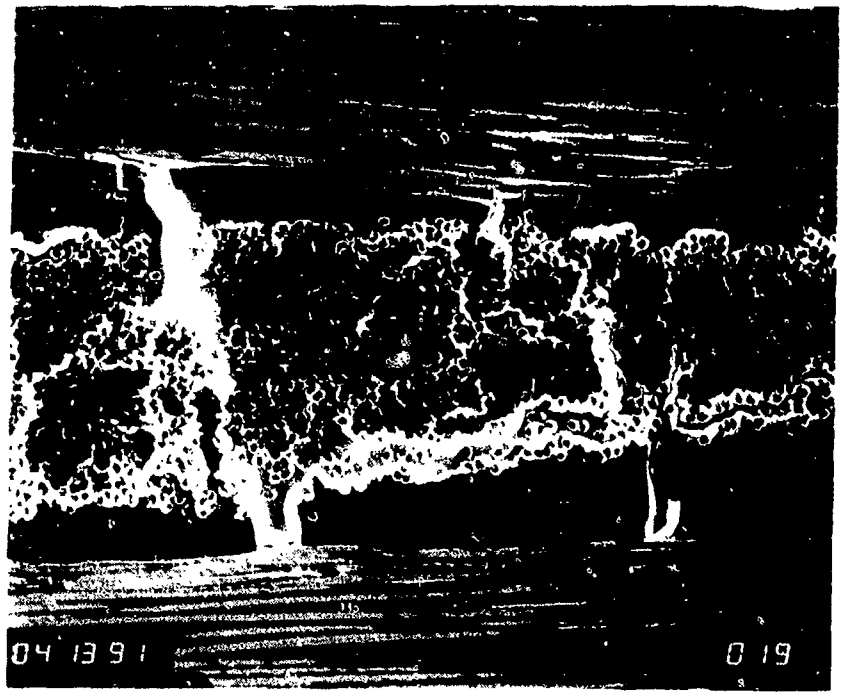


Figure 13. Optical Photograph Showing Oxidized Carbon-Carbon Specimen Impregnated with Fluorescent-Tagged Resin to Highlight Oxidized Areas.

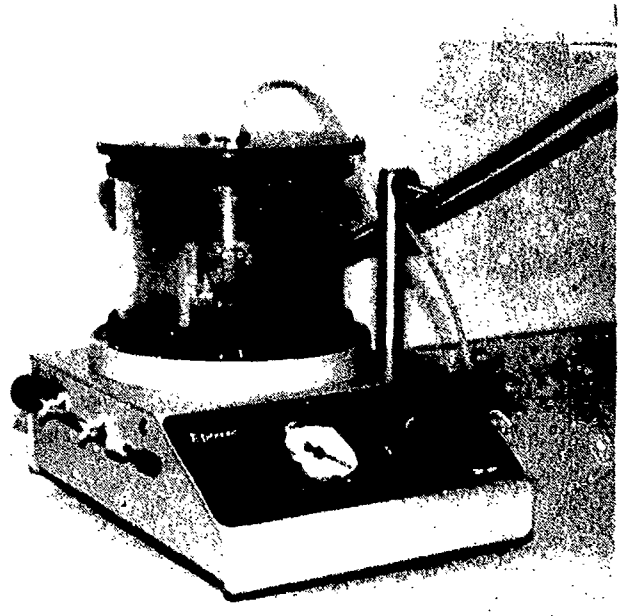


Figure 14. Struers Vacuum Impregnation Chamber.

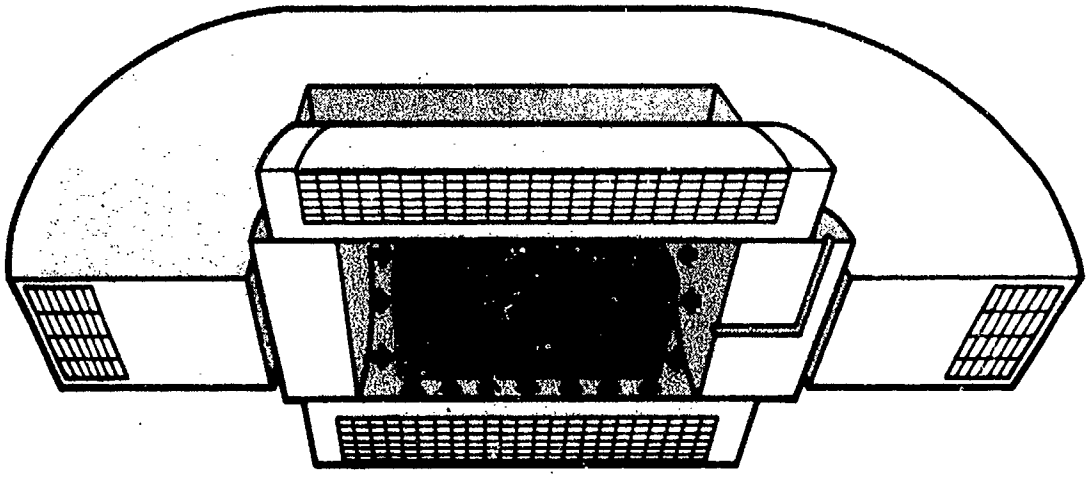


Figure 15. Drawing of Hot Isostatic Press Chamber Used to Impregnate Carbon-Carbon Composites with Resin or Low Melting Alloy.

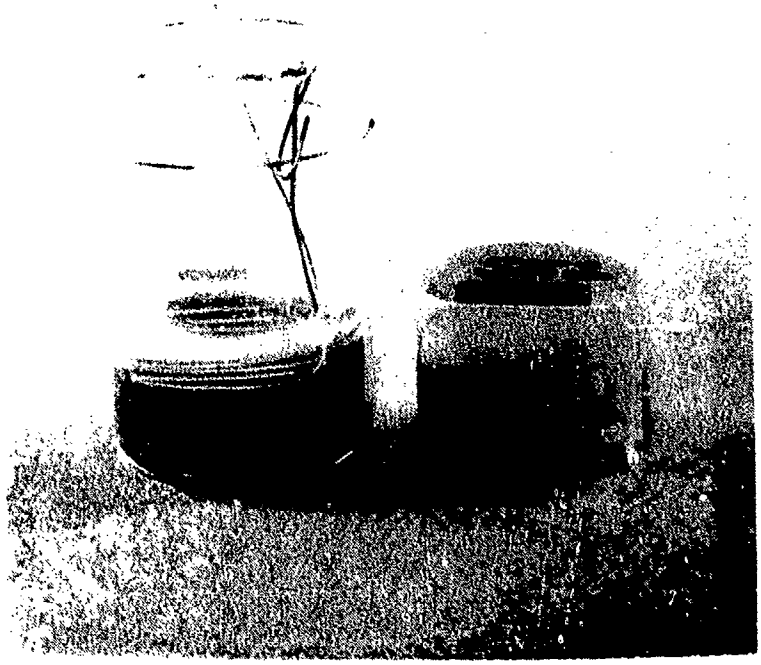
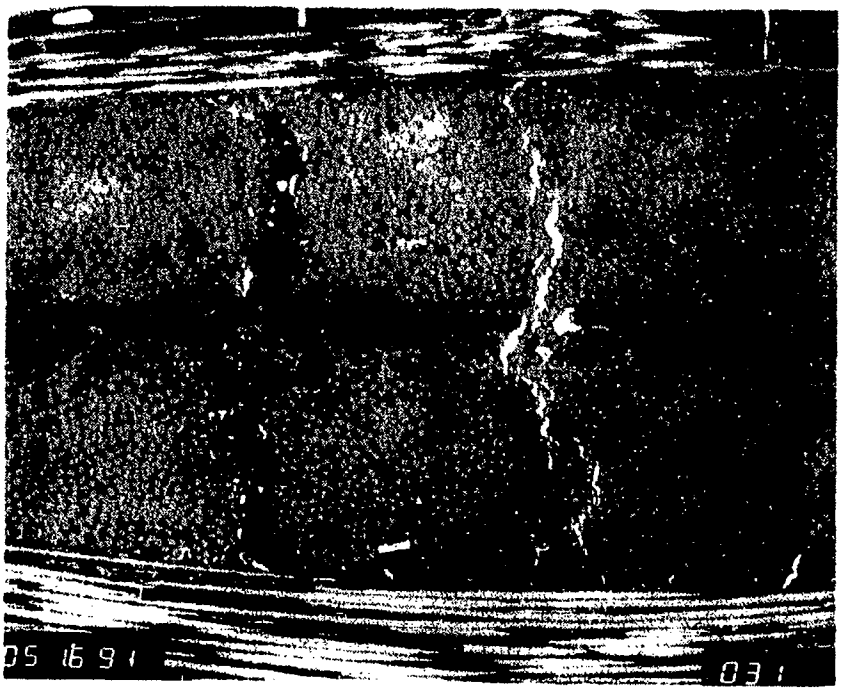
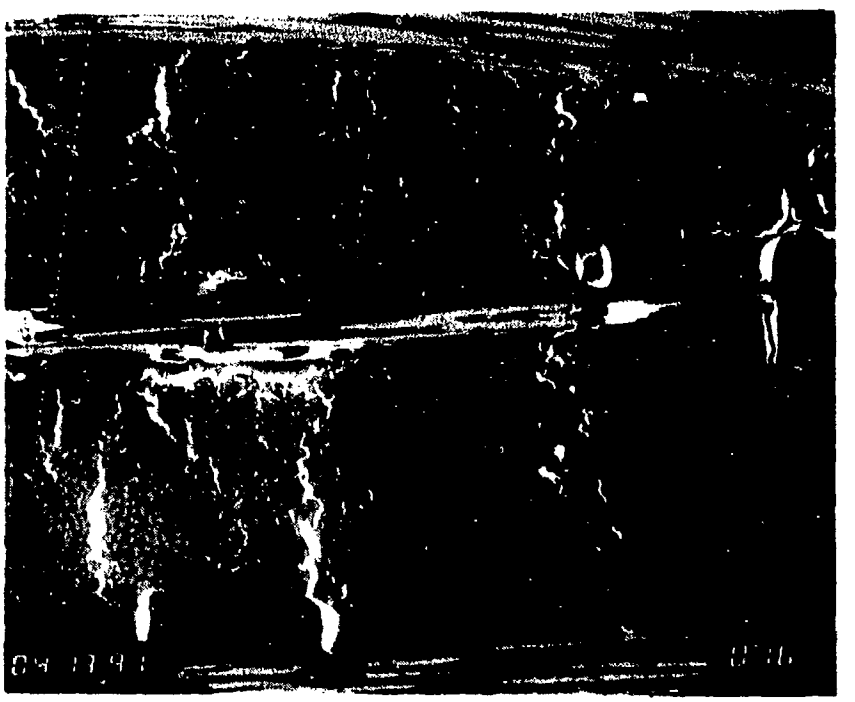


Figure 16. Photograph of Containment Vessel Used to Pressure Impregnate with Resin and Plug Containing Resin-Impregnated Carbon-Carbon Specimen.

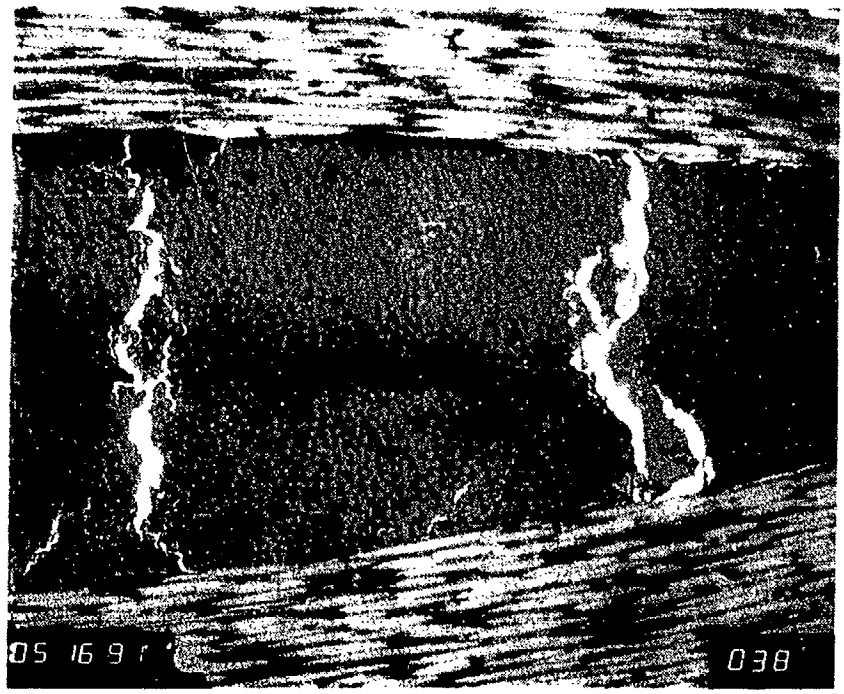


(a)



(b)

Figure 17. Optical Photographs Showing Partial Infiltration of Fluorescent-Tagged Impregnation Resin (200X).



(a)

(b)

Figure 18. Optical Photographs Showing 100% Infiltration of Fluorescent-Tagged Resin (200X).

impregnated by the epoxy) will also be distinguishable. This technique can be used to detect microscopic cracks and to determine pore density of carbon-carbon composites as processed or after thermal cycling to evaluate oxidation resistance. The enhancement of the cracks and pores with a fluorescent dye can complement image analysis as a tool to characterize this material.

4.4 Impregnation with a Low Melting Alloy

An effort was made to impregnate the open void space in a carbon-carbon composite with a low-melting alloy (metal) of high atomic number to enhance the contrast between carbon (either fiber or matrix) and void space in an electron microscope. A metal impregnant may also lend itself to a variety of nondestructive techniques such as eddy current and computed tomography in order to assess composite quality.

A carbon-carbon composite specimen with dimensions of 0.75-inch x 0.75-inch x 0.375-inch was secured at the bottom of a 30-ml Pyrex beaker (Figure 19) with a length of steel wire to prevent it from rising to the surface when submerged in molten metal. Approximately 70 grams of low-melting alloy (LMA158) were solidified in a 20-ml Pyrex beaker so that the resulting cylindrical plug could be placed over the composite specimen in the 30-ml beaker. This beaker was then placed in the Hot Isostatic Press (HIP). The HIP was purged twice with argon and then evacuated to 0.8 Torr for 10 minutes. The temperature was increased to 120°C and held for one hour to completely liquefy the alloy, thus submerging the specimen. A pressure of 1 ksi was then applied for 30 minutes to force the liquid metal into the open cavities of the specimen. The HIP was then cooled slowly over a four-hour period under pressure. Two sections were cut from the specimen and examined in an optical microscope to determine the extent of metal infiltration. Although most of the cavities were filled with metal, a few isolated cavities appeared only partially impregnated (Figure 20). Darkfield microscopy was attempted to determine if the metal-filled cavities were enhanced (Figures 21 and 22). It was difficult to distinguish between voids and metal-filled cavities using this method since both were enhanced to some degree. A second specimen was impregnated using the above procedure, but the applied pressure was increased to 5 ksi. Optical microscopy indicated 100 percent impregnation (Figure 23) of the open cavities in this case.

In addition to optical microscopy, metal-impregnated specimens were also examined using scanning electron microscopy (SEM) to determine if the metal would enhance the microstructural voids and cracks. SEM was conducted in the secondary and backscattering (compositional and topographical) modes. Due to the high atomic number of the impregnating

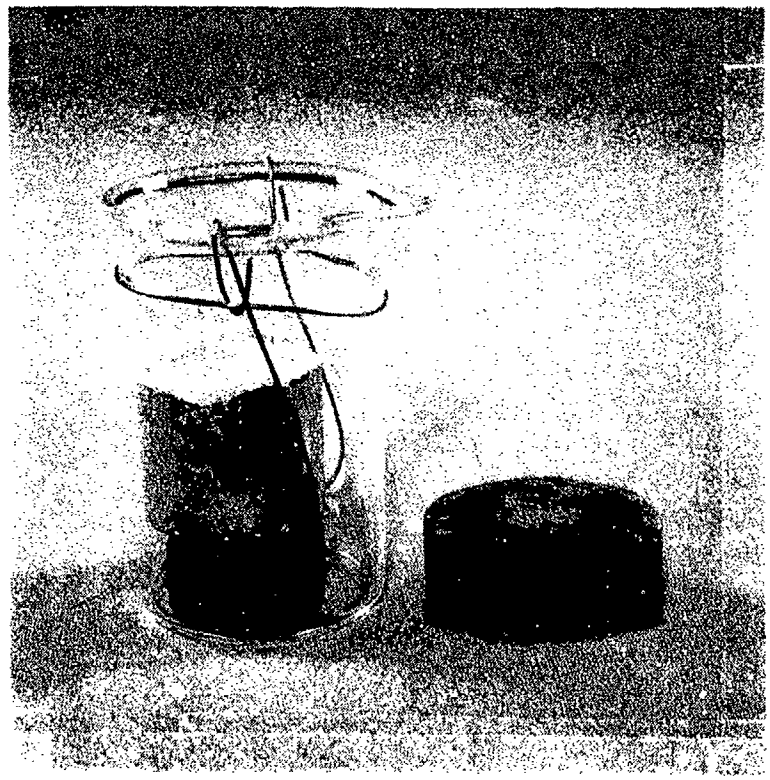


Figure 19. Photograph of Containment Vessel Used to Pressure Impregnate with Low Melting Alloy and Plug Containing the Impregnated Carbon-Carbon.

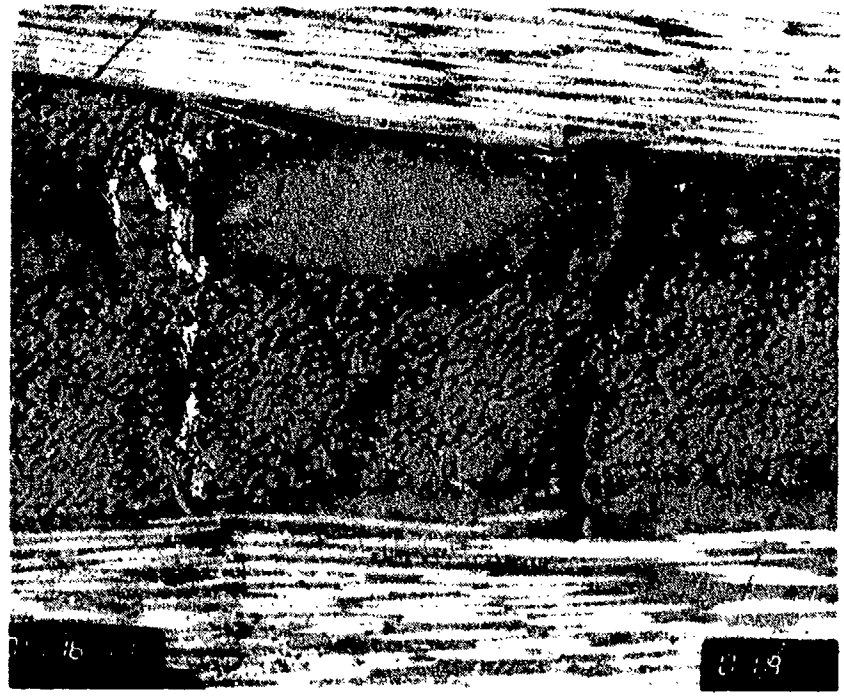


Figure 20. Optical Photograph Showing Partial Infiltration of Metal (200X).

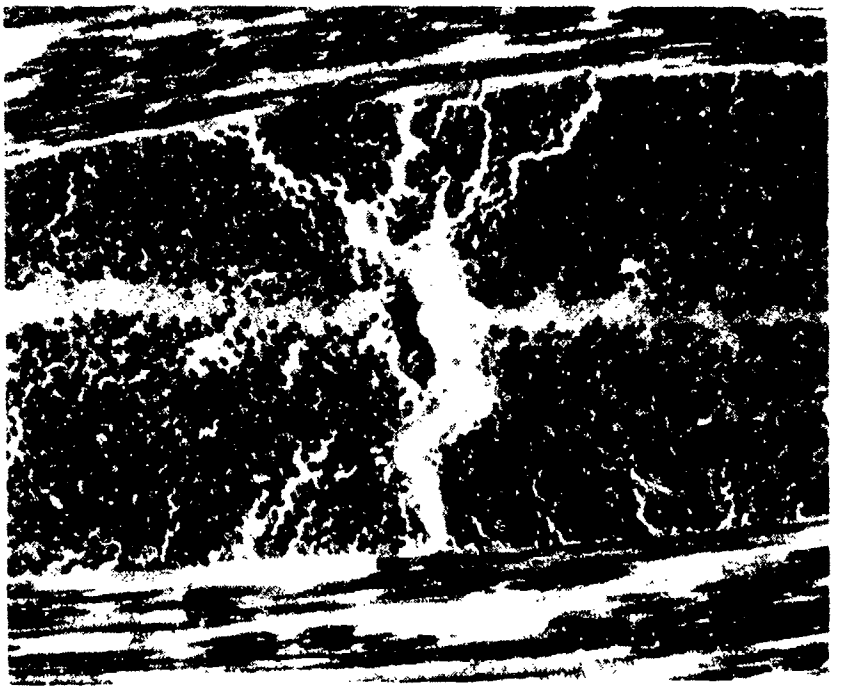


Figure 21. Darkfield Optical Photograph Showing Enhancement of Metal-Filled Cavities (200X).

Figure 22. Darkfield Optical Photograph Showing Enhancement of Metal-Filled Cavities (50X).

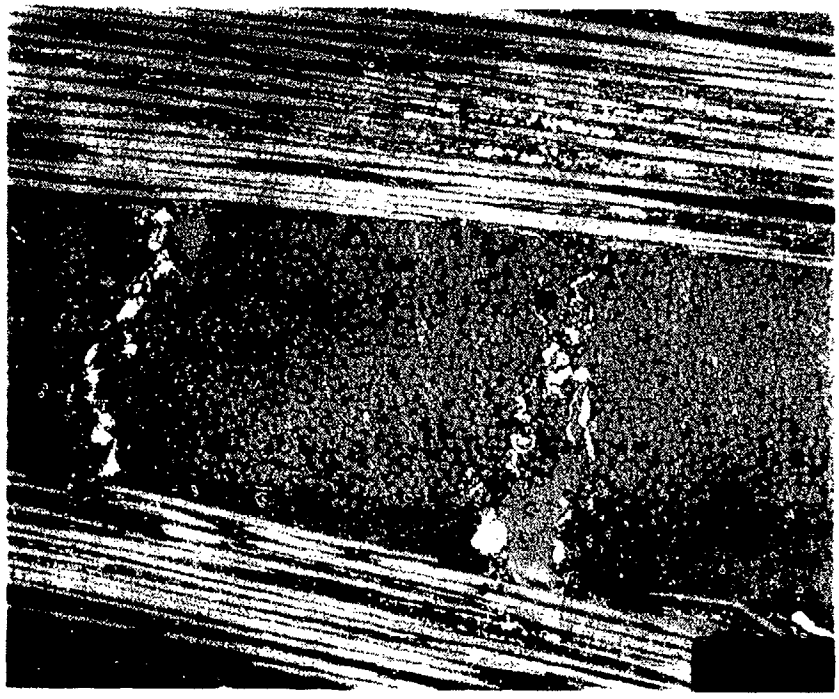


Figure 23. Optical Photograph Showing 100% Metal Infiltration (200X).

microstructural voids and cracks. SEM was conducted in the secondary and backscattering (compositional and topographical) modes. Due to the high atomic number of the impregnating metal, filled cavities were greatly enhanced when viewed in the compositional mode (Figure 24). In the topographical mode (Figure 25), metal-filled areas are also enhanced along with some surface artifacts. The secondary mode (Figure 26) also enhances the metal; however, the contrast between the LMA158 and the surrounding areas was not as apparent. Neither computed tomography nor eddy current measurements were considered useful at this time in evaluating the porosity with metal-impregnated specimens.

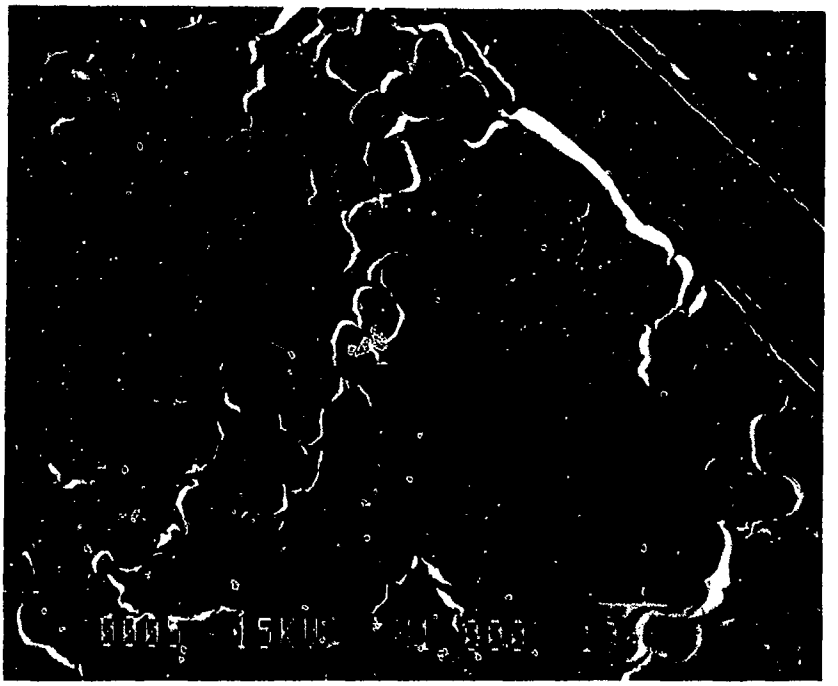


Figure 24. SEM Taken in Compositional Mode Showing Enhancement of Metal Infiltration into Open Porosity (1000X).

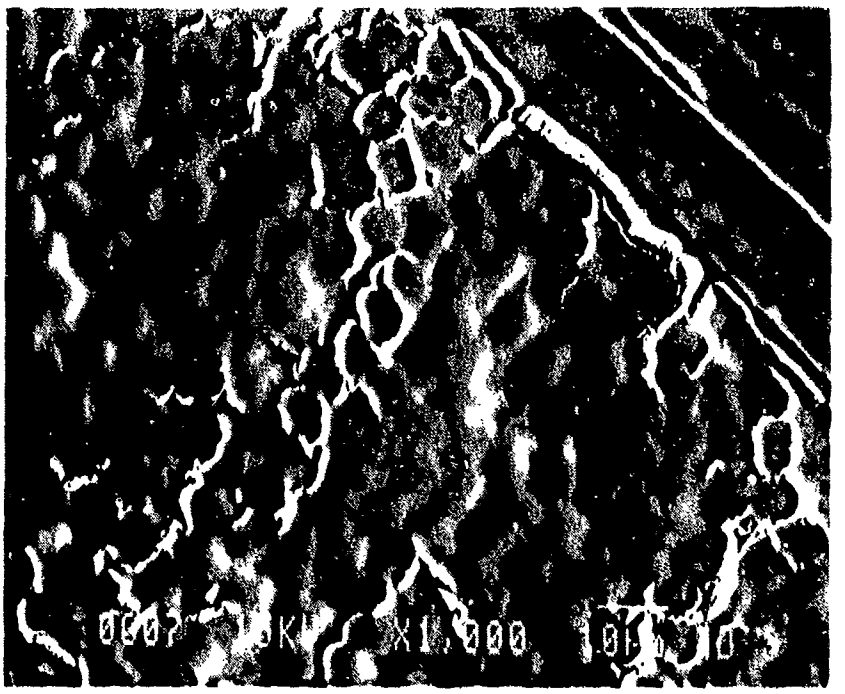


Figure 25. SEM Taken in Topographical Mode Showing Enhancement of Metal Infiltration into Open Porosity (1000X).

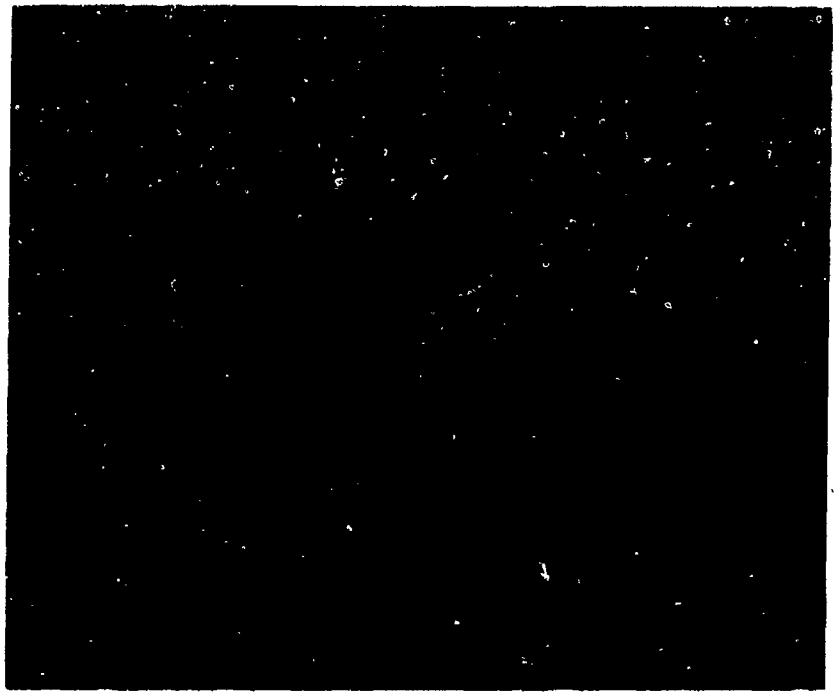


Figure 26. SEM Taken in Secondary Mode Showing Enhancement of Metal Infiltration into Open Porosity (100X).

5. SPECIMEN MOUNTING TECHNIQUES

Prior to microstructural analysis a composite specimen is usually encapsulated in a resin plug and polished. Such mounting provides edge retention and prevents fiber tear-out during polishing which would otherwise damage the polished surface. Due to the high porosity and somewhat fragile nature of carbon-carbon composites, it was considered necessary to establish mounting techniques and media that would suit this unique material. Three resin systems from Buehler Products were evaluated for mounting carbon-carbon specimens, a two-part cold mounting system, and two compression molding systems. The cold mounting system cures in air at room temperature in approximately 30 minutes. Both compression molding materials are epoxy resins that require heat and pressure for curing.

The cold mounting epoxy, Epo-Kwick, was the softest of the three resins evaluated. If possible it is important to match the hardness of the mounting material with that of the specimen to be polished. A mount which is significantly softer than the specimen will abrade faster and fail to protect the surface to be polished. This may have been the case with this cold mount, since it was difficult to polish carbon-carbon specimens completely free of surface scratches. One method of increasing the hardness of the mounting material is the addition of a finely-ground alumina powder. The entrapment of air bubbles which adhere to the specimen surface during the encapsulation process must be avoided. These voids in the cured product trap debris and polishing compounds and are carried from one step to the next. Occasionally clips are employed to hold specimens in place during the encapsulation. In such cases plastic clips are preferred to those of stainless steel, since the former better match the properties of the mounting resin.

The two compression molding resins require both heat and pressure for curing and are processed in an automated pneumatic press. The first of these mounts evaluated was Epomet. This epoxy compound is processed at 150°C and at pressures between 1500 to 2500 psi. This material produces a flat, void-free plug ready for grinding and polishing. With this mount it was easier to produce a scratch-free polished surface when mechanically polished than with the Epo-Kwick cold mount system. This mounting material is harder than the cold mount and, unlike the latter, not susceptible to distortion when secured in the specimen holder of the mechanical polisher. This distortion can reduce the flatness of the surface to be polished which in turn influences its finished quality. The second thermosetting mount evaluated was Konductomet I, also an epoxy material containing a carbon filler, thus making the cured plug conductive. The conductive plug and mounted specimen can be examined in the SEM without the need for

additional preparation steps such as sectioning, remounting, and sputter coating. The quality of the polished surface resembles that obtained with the Epomet mount.

Some specimen alteration such as crushing was observed using the compression molding technique. This was mostly detected on long, thin carbon-carbon specimens with large aspect ratios. In such cases the pressure was reduced to 1500 psi which eliminated the problem. If compression molding is selected for mounting carbon-carbon composites, it is suggested that it be conducted along with vacuum impregnation when possible. This will enhance the resistance of the specimen to damage under applied pressure and is especially applicable in the case of fragile oxidized carbon-carbon specimens.

6. GRINDING AND POLISHING TECHNIQUES

The surface of a mounted (or unmounted) carbon-carbon composite specimen must be ground and polished before it is microscopically examined. Grinding can be accomplished by hand on a sanding block or mechanically. For carbon-carbon composites successive use of 240-, 320-, 400-, and 600-grit silicon carbide papers is recommended, although the first two are optional. A variety of approaches can be employed in these preparation steps. As stated in the Metals Handbook concerning polishing, "Experience, common sense, and trial and error should determine the best approach for a specific application. The goal is to produce a high-quality, scratch-free specimen with minimum topographical variation [3]." There are several ground rules to follow when polishing any composite. These include the generous use of distilled water, oil or solvent to lubricate and to carry debris away from the polished surface; avoiding excessive pressure on the specimen and grinding wheel speed with mechanical polishing methods, to avoid fiber tearout; flushing the specimen surface between polishing steps to remove debris; and changing the polishing materials between each job to prevent scratching or contamination from a previous job.

Carbon-carbon composites often have constituents with a mismatch of properties such as metal surface coatings or oxidation inhibitors within the internal structure of the composite. The hardness of these constituents can vary greatly, and special care may be required to avoid damaging them during preparation steps. The high porosity of most carbon-carbon composites presents an additional problem. If polished as received, the cavities will get considerably enlarged. In addition there will be little edge retention and fibers may be pulled out. It is recommended that the specimen cavities be impregnated with a supportive resin prior to polishing. To assess different polishing techniques, media, and equipment, specimens were polished in-house on various pieces of metallurgical polishing equipment and also submitted to outside sources.

The workhorse for specimen polishing concerning this study is the Buehler Maximet specimen preparation system. This machine can polish 24 standard plugs at a time or larger specimens up to a length of 11 inches and two-inch thickness. Grinding and polishing are transmitted to the specimen holder via the vertical load Maximet head. The heat can be rotated in two directions and also can be programmed to sweep in one of three different arcs. The platen speed can be adjusted at 100 and 200 rpm. There is a wide range of abrasive materials available for use with this machine. The Maximet is a versatile machine that gives the operator many control choices and produces polished surfaces of consistent quality. The machine can handle

rough grinding as well as fine polishing by simply changing the abrasive paper or polishing surface and using the appropriate polishing medium.

Several different polishing techniques developed for carbon-carbon using the Maximet are listed below with their sequential steps. These techniques were modified when necessary to accommodate the many different constituents often found in carbon-carbon composites. Modifications were also necessary when a different mounting material was used. Each technique produced satisfactory results. Buehler polishing products were used in most cases.

Technique 1. Polishing Cold-Mounted Specimens (First Procedure) Hitco 8A Carbon-Carbon

(1) Specimen cast in epoxy mounting material
(2) 320-grit silicon carbide paper, 5 psi, 2 minutes
(3) 400-grit silicon carbide paper, 5 psi, 2 minutes
(4) 600-grit silicon carbide paper, 5 psi, 2 minutes
(5) 3-micron diamond paste on Texmet cloth, Metadi lube, 5 minutes
(6) 1-micron diamond paste on nylon cloth, Metadi lube, 5 minutes
(7) Mastermet polishing suspension, Chemomet cloth, water lube
(8) Clean with Ivory soap, rinse with water and ethanol
(9) Vibratory polishing in Mastermet on Microcloth for 60 minutes, with added weights to decrease polishing time

Technique 2. Polishing Cold-Mounted Specimens (Second Procedure) Hitco 8A Carbon-Carbon

(1) Specimen cast in epoxy mounting material
(2) 400-grit silicon carbide paper, Ivory soap, water, 2 minutes
(3) 600-grit silicon carbide paper, Ivory soap, water, 2 minutes
(4) 3-micron diamond paste, Metadi 3-micron diamond compound, Metadi fluid, 8 minutes, Texmet cloth
(5) 1-micron diamond paste, Metadi 1-micron diamond compound, Metadi fluid, 8 minutes, Texmet cloth
(6) Masterpolish with 0.06 alumina abrasive, Chemomet cloth
(7) Vibratory polishing in Mastermet, Microcloth

Technique 3.Polishing Compression-Molded Specimens Hitco 8A Carbon-Carbon

(1) Specimen compression molded with Epomet mounting material
(2) 320-grit silicon carbide paper, 5 psi, 2 minutes
(3) 400-grit silicon carbide paper, 5 psi, 2 minutes
(4) 600-grit silicon carbide paper, 5 psi, 2 minutes
(5) 6-micron diamond paste on Texmet cloth, Metadi lube
(6) 3-micron diamond paste on Texmet cloth, Metadi lube
(7) 1-micron diamond paste on Texmet cloth, Metadi lube
(8) Mastermet polishing suspension, Chemomet cloth, water lube
(9) Vibratory polishing in Mastermet on Microcloth for 60 minutes, with weights to decrease polishing time

The above mounting techniques were described in Section 5.

A Nikon Epiphot, inverted microscope, in conjunction with a Nomarski filter, was used to assess the quality of the polished surfaces. The polished surfaces of the compression-molded specimens had fewer scratches and less fiber pullout than those of the cold-mounted specimens. This is attributed to the harder mounting material of the compression molded plugs which better matches the hardness of the carbon-carbon composite specimen.

Carbon-carbon composites frequently contain nonreactive or reactive boron particles in the matrix to inhibit oxidation at elevated temperatures. The boron is soluble in water unless converted to boron oxide at high elevated processing temperatures. A nonaqueous polishing technique was developed if boron reactivity with water is a concern.

All boron inhibited carbon-carbon specimens for this study were compression molded with Epomet resin following the various steps detailed below.

Technique 4.Polishing Inhibited Carbon-Carbon in an Aqueous Medium

(1) Metadi 45-micron diamond (water based), #8 Metlap Wheel, Ivory soap lube, 5 psi head pressure
(2) 15-micron diamond paste, 6-micron Metadi diamond slurry lube, Ivory soap, Texmet cloth (perforated)
(3) 6-micron diamond paste, 6-micron Metadi diamond slurry (water based), Texmet cloth (nonperforated)

(4) 1-micron diamond paste, Metadi lube, Texmet cloth
(5) 0.3 Alumina slurry, Chemomet cloth

Technique 5.Polishing Inhibited Carbon-Carbon in a Nonaqueous Medium

(1) 45-micron diamond paste mixed with Hyprez lubricant from Engis Corporation, #8 lapping wheel, 5 psi head pressure, Beta lube
(2) 15-micron diamond paste mixed in Hyprez lubricant, Beta lube, #8 lapping wheel, 5 psi
(3) 3-micron diamond paste mixed in Hyprez lubricant, Beta lube, #8 lapping wheel, 5 psi
(4) 1-micron diamond paste applied to surface of Texmet cloth, and 1-micron diamond paste mixed in Hyprez lubricant, 5 psi
(5) 0.5-micron diamond paste applied to Chemomet cloth, Hyprez lubricant added to cloth, Beta lube, 5 psi

Surface coatings, such as silicon carbide, are often employed to protect the carbon-carbon substrate from oxidation. This material is significantly harder than the carbon-carbon substrate and does not polish as easily. This coating must first be taken down with a 45-micron diamond paste applied to a sanding block containing 600-grit silicon carbide paper. This is followed by polishing with a 15-micron paste and a 3-micron paste on a nylon cloth. A microcloth is then used with a 3-micron alumina slurry followed by a 0.05 alumina slurry. An oil or water lubricant is recommended. The alumina removes fine scratches that the diamond paste will not. This polishing procedure was accomplished by hand on polishing wheels set at low speed.

Technique 6.Grinding and Polishing Procedure Accomplished on the Buehler Ecomet 3 Grinder/Polisher with an Automet 2 Power Head

This work was accomplished on Hitco 8A material at the Buehler labs using cold-mount plugs.

Grinding: 320-grit SiC, 5-lb load, 120 rpm, 2.5 minutes
400-grit SiC, 5-lb load, 240 rpm, 2 minutes
600-grit SiC, 5-lb load, 240 rpm, 2 minutes

Rough Polish: Texmet cloth, lapping oil, 9-micron diamond paste, 5-lb load, 120 rpm, 2.5 minutes
Texmet cloth, lapping oil, 3-micron diamond paste, 5-lb load, 120 rpm, 3 minutes

Final Polish: Chemomet cloth, Masterpolish, 10-lb load, 120 rpm, 3 minutes (contra head rotation on this step)

Buehler advised that scratches may be removed in a shorter time by employing a head pressure of 10 pounds instead of 5 pounds in the final polishing step. With an automatic polisher, a contra (against the wheel) head rotation is conventionally used on the rough and final polishing. To minimize fiber pullout on the carbon-carbon composite sample, a complementary (with the wheel) head rotation was used on all steps but the final polish. This is recommended for many materials where special care is needed.

Technique 7.

Carbon-carbon 8A specimens were sent to Struers, Inc. to evaluate vacuum impregnation with a fluorescent-tagged resin (Epofix) and polishing. Listed below is the polishing procedure used on Struers equipment. The plugs were cold mounted.

Automatic Grinding	**PG1**	**FG1**	**FG2**	**FG3**
Equipment	Abrapol-2	Abrapol-2	Abrapol-2	Abrapol-2
Disc/Cloth	Paper	Paper	Paper	Paper
Abrasive	SiC	SiC	SiC	SiC
Grit/Grain	320	800	2400	4000
Speed (rpm)	300	300	300	300
Load (N)	100	100	100	100
Lubricant/Dosing	water	water	water	water
Time	until plane	30 sec	30 sec	1 min

Automatic Polishing	Dp 1	Dp 2
Equipment	Abrapol-2	Abrapol-2
Disc/Cloth	Pan-W	OP-Chem
Abrasive	Diamond	OP-S
Grit/Grain	3 microns	0.04 micron
Abrasive Dosing	150	150
Speed (rpm)	150	100
Load (N)	0	0
Lubricant/Dosing	Blue #2	--
Time	5 min	3 min

7. MICROSCOPIC EXAMINATION

7.1 Optical Microscopy

Both inverted and upright brightfield microscopes were utilized to study the microstructure of carbon-carbon composites. Two inverted microscopes were utilized, a Nikon Epiphot and a Reichert-Jung MeF3-A, the latter being equipped with a 400-W xenon light source for fluorescence microscopy. A Microphot-FXL upright microscope with a 100-W mercury light source was also employed for fluorescence microscopy. Optical microscopy was used to examine composite quality, the number of plies, ply character, thickness variations, resin-rich areas, delaminations and determination of crack/void densities.

Darkfield illumination was briefly discussed in Section 4.4 when the technique was used to enhance the metal-filled porosity. In this case darkfield illumination was used to detect fluorescence-tagged resin in vacuum-impregnated carbon-carbon composites before a fluorescence microscope was available.

The principle of darkfield illumination is the formation of a hollow cone of light whose apex lies in the plane of the specimen. When the light is carefully focused at the plane of the object but no object is present, the hollow cone of light passed through the condenser produces no illumination in the microscope because the objective is inside the dark base of the hollow cone. When a specimen is present, the light is deviated, or scattered, into the objective by the structures on the specimen. A bright image of these details is then visible against a dark background. Because of the high contrast of the image, the system is capable of detecting extremely small particles [2]. This may prove useful when studying oxidation inhibitors which are present in many carbon-carbon composites.

The cavities filled with the fluorescence-tagged resin were enhanced using darkfield illumination (Figure 27), but unfilled cavities were also enhanced nearly as well. If 100 percent resin infiltration can be confirmed with brightfield techniques, then brightfield illumination could prove beneficial. Similar results were obtained with metal-impregnated carbon-carbon composites except that the metal was enhanced to a greater degree with darkfield illumination than the fluorescent-tagged resin.

Polarized light microscopy was briefly investigated to identify various constituents in carbon-carbon composites. As stated in the Metals Handbook, "The birefringent (optically anisotropic) nature of the graphitic matrix of carbon-carbon composites can be used under cross-polarized light to identify and determine the structure of each microconstituent. Polarized light

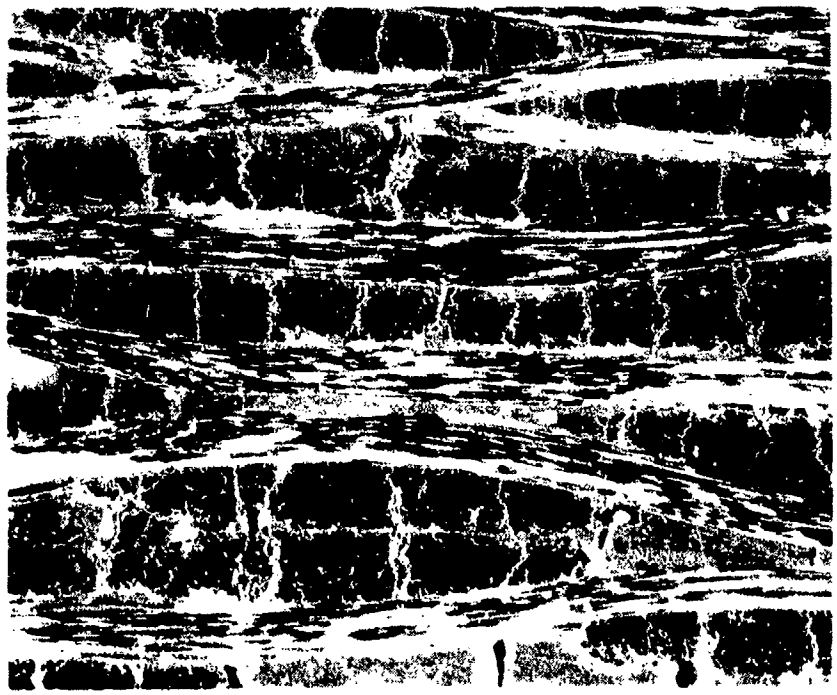


Figure 27. Darkfield Optical Photograph Showing Enhancement of Resin (50X).

may reveal the structure of the matrix, pore morphology, microcrack morphology, degree of densification, and fiber volume fraction. In bidirectional composites, ply orientation and spacing as well as the extent of any anomalies can also be determined [3]." It would be beneficial to evaluate this technique further in the future.

7.2 Confocal Microscopy

Both carbon-carbon composites and reticulated vitreous carbon foams were submitted to Sarastro, Inc. to evaluate Confocal Microscopy, a fluorescence-based technology. The following explanation describes the basic concept behind confocal microscopy. Laser light is reflected by a dichroic beam-splitter into a scanning unit which moves the beam back and forth in a raster pattern, and then is brought to a fine point of focus in the specimen by a high numerical aperture objective. Emitted fluorescence passes back through the same optics, through the scanner and dichroic, and is brought to a final focus at an aperture which then activates a detector. Light which is above or below the specimen plane of focus is imaged long or short of the aperture, and light coming from either side is stopped by the aperture, producing very thin optical sections of high contrast. The technique can extract images from different depths. It allows the microscope

to penetrate a semitransparent fluorescence specimen and extract images that, with regular equipment, would be completely blurred by light from regions above or below the inspected plane. A stack of images representing consecutive optical sections is produced and stored in digital form. This stack constitutes the basis for reconstructing and visualizing the three-dimensional structure of a specimen. This technique can be used on specimens that, when appropriately illuminated, return either reflected or fluorescent light [4]. The carbon-carbon composite images clearly show the weave pattern of the cloth reinforcement at surface and possibly subsurface levels. However the resolution is very poor. The carbon foam images (Figure 28) yielded similar results. To date, more useful information has been obtained with electron microscopy and optical microscopy techniques. The three-dimensional reconstruction does not approach the quality that was desired. However further investigation may be needed before this technique is ruled out.

7.3 Scanning Electron Microscopy

The use of electron microscopy to detect both metal and resin-infiltrated carbon-carbon was discussed in previous sections. Additional information is offered in the following section.

A Jeol JSM-840 scanning electron microscope was used to examine resin-impregnated and metal-impregnated carbon-carbon composites in both secondary and backscattering modes. The secondary mode is commonly used to obtain an image of the specimen. The backscattering mode has two image options - compositional image and topographic image. The compositional image shows the atomic number contrast of the specimen, while the topographic image shows the topographic appearance of the specimen surface (refer to Figures 24, 25, and 26 in Section 4.4). Figure 29 illustrates the schematic principle of backscattered electron microscopy for obtaining compositional and topographic images.

The filled cavities of the resin-impregnated specimens were enhanced; however, filled cavities were also enhanced, albeit to a lesser degree, due to surface charging, since the specimens were examined uncoated. Sputter coating with a conductive layer will reduce surface charging but will also mask the enhancement of the resin-filled cavities. The technique most preferred to evaluate porosity in carbon-carbon composites is through impregnation with a resin tagged with a fluorescent dye. Sectioned and polished specimens can then be examined in a fluorescence microscope. The backscattering mode proved to be useful when examining coated carbon-carbon composites. The different layers of the Si/SiC coating deposited on the surface of the carbon-carbon substrate via chemical vapor deposition are more defined in the backscatter

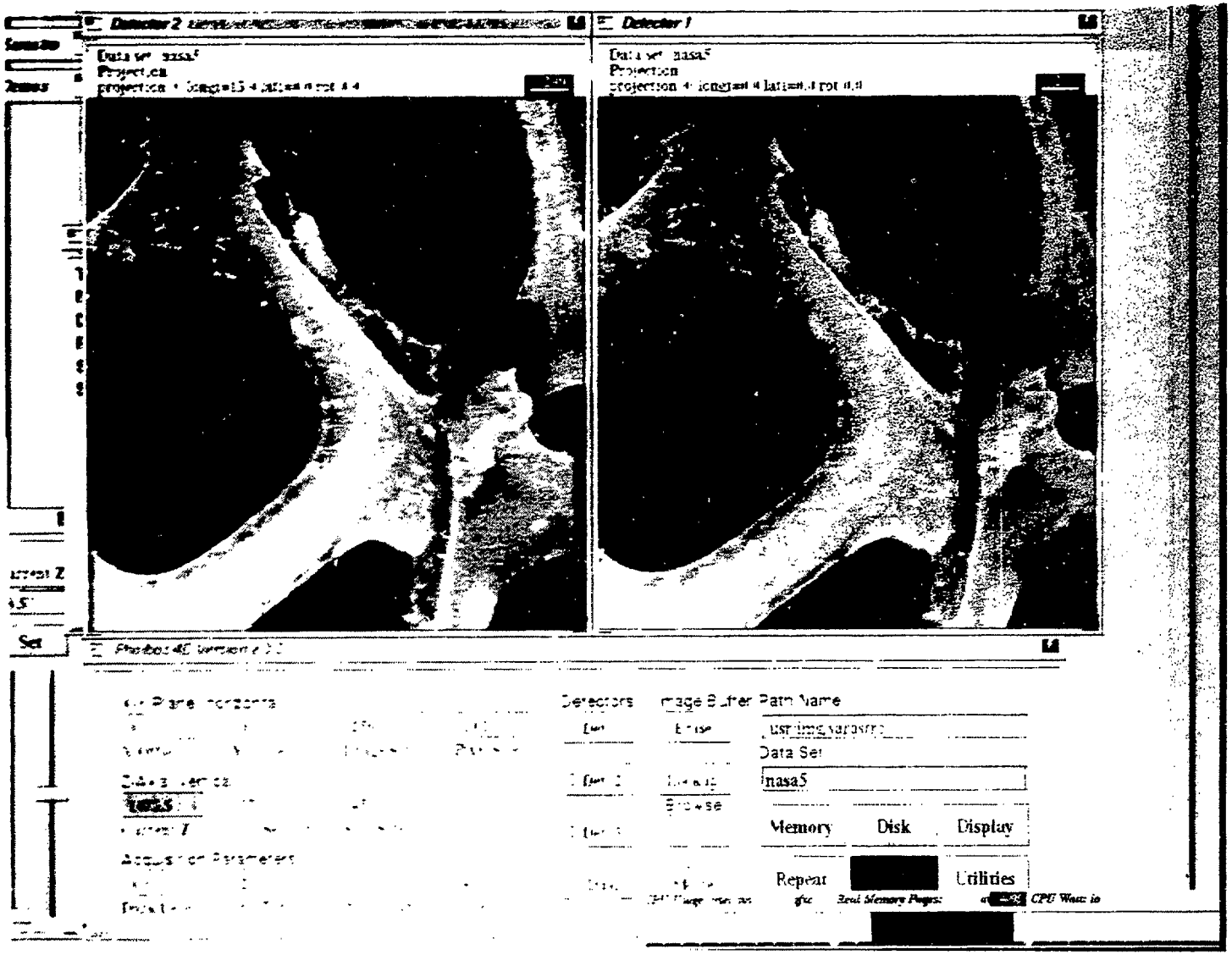


Figure 28. Confocal Image of Reticulated, Vitreous Carbon Foam.

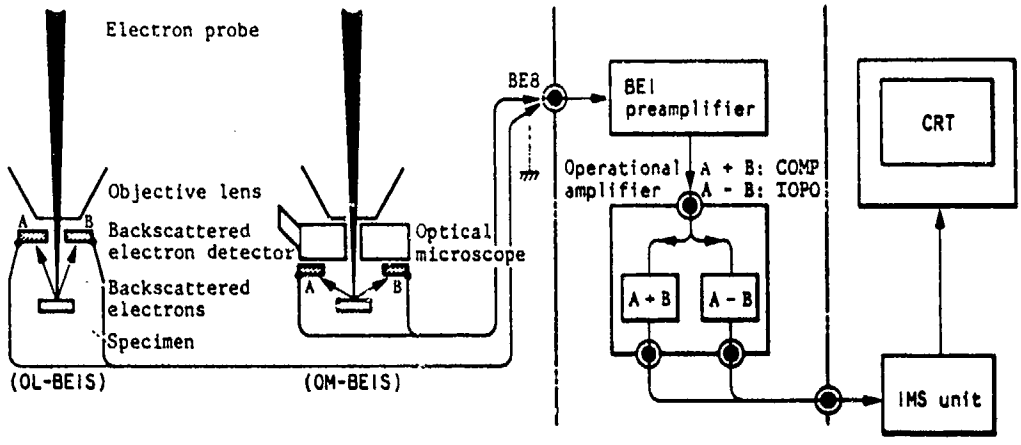
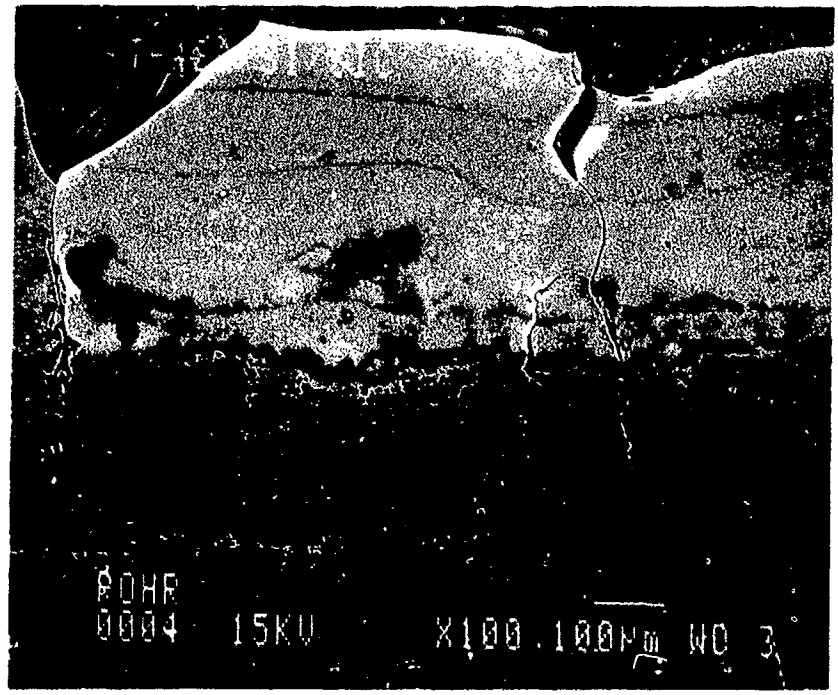


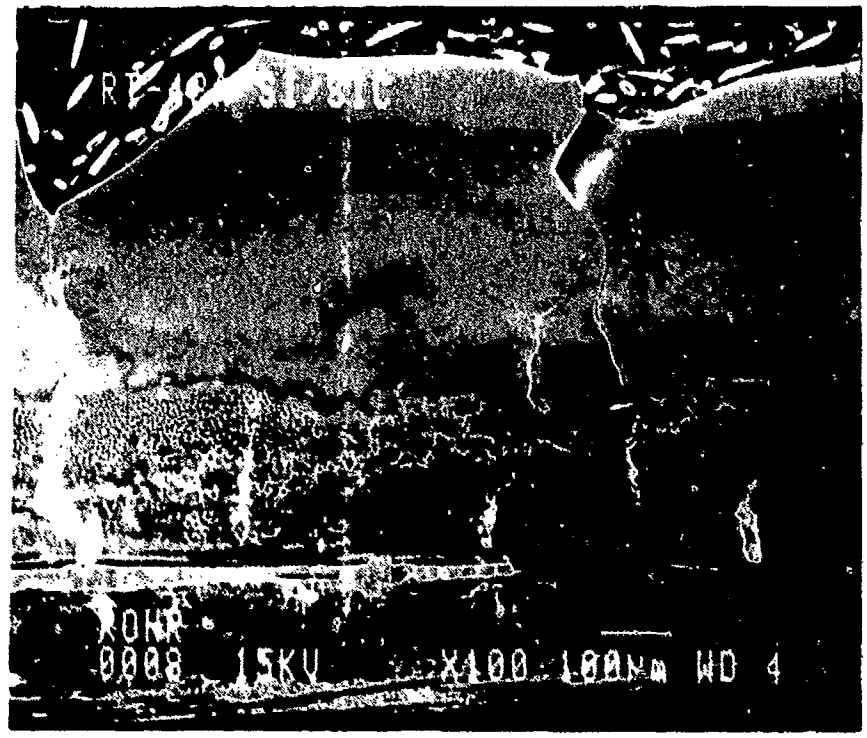
Figure 29. Schematic Principle of Backscattered Electron Microscopy for Obtaining Compositional and Topographic Images.

compositional mode (Figure 30A) when compared to the secondary mode (Figure 30B). This is attributed to atomic number contrast between the elements of the different layers.

Polished carbon-carbon composites or as-sectioned specimens reveal little microstructure when observed under SEM. Specimens which exhibit little reflectivity can be etched to produce sufficient image contrast and thereby reveal the microstructural features. Ion bombardment was employed to etch the composite specimens. Also called cathodic vacuum etching, this technique produces structural contrast by selective removal of atoms from the specimen surface. This is accomplished by using high-energy ions accelerated by voltages of 1 to 10 kV. Individual atoms are removed at various rates depending on the microstructural details such as crystal orientation of the individual grains, grain boundaries, etc. [5]. Both xenon and argon gas etching were evaluated at Aerospace Corporation and Gatan Corporation, respectively. Xenon etching is preferred since it produces the fastest etch. All specimens were polished prior to being etched so that the etched area could be easily discerned and comparisons made with the surrounding nonetched area.



(a)



(b)

Figure 30. SEMs Showing Increased Enhancement of Si/SiC Layers on Coated Carbon-Carbon Composite (100X). (a) Backscatter Image (Compositional Mode); (b) Secondary Image

At Aerospace Corporation xenon etching was conducted on equipment built in-house. Etching was conducted at 4-kV-positive charge for 30 minutes. SEM micrographs show various degrees of relief and microstructural detail in matrix/pitch fiber areas on an RX715V carbon-carbon composite (Figures 31A and 31B). Densified layers and chemical vapor infiltration phases can easily be discerned in the etched samples. Figures 32A and 32B also illustrate the difference between unetched and etched microstructures using SEM on a Noveltex CVD/PAN carbon-carbon composite. Figure 33 illustrates the xenon etched surface of a PAN fiber in the longitudinal direction.

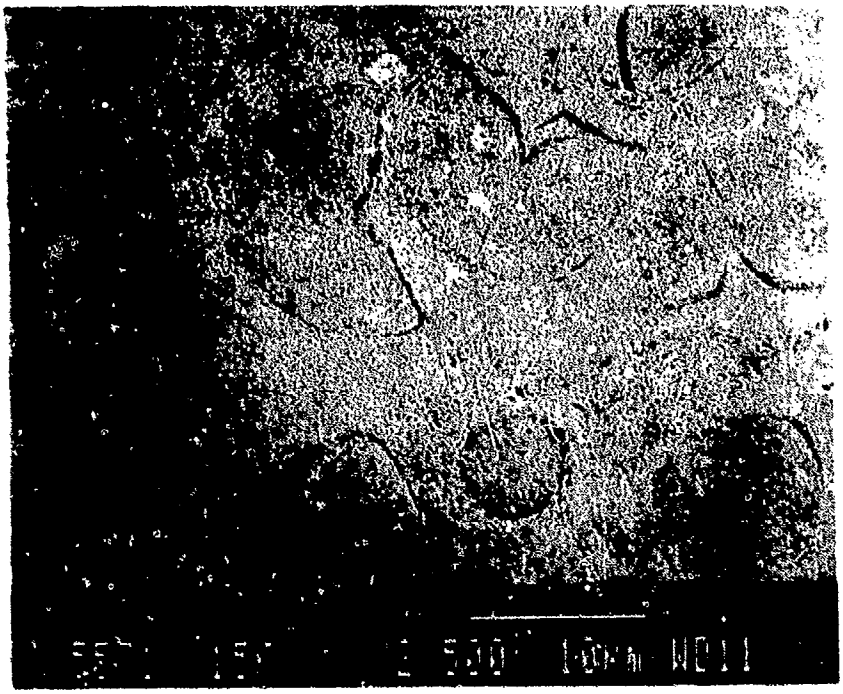
Gatan Corporation modified an ion mill commonly used for TEM specimen preparation and etched specimens in argon for one hour at 7 kV and 0.5 mA, at an incident angle of 20 degrees. Microstructural detail is evident at the fiber ends with possibly an excessive amount of relief (Figure 34). Further optimization of the experimental parameters is required. It should be kept in mind that this technique of improving the contrast alters the characteristics of the surface being examined.

7.4 Cathodoluminescence Microscopy

Cathodoluminescence (CL) microscopy, a technique used to study ceramics, is being evaluated to examine carbon-carbon composites. The cathodoluminescence microscope utilizes a beam of electrons produced by a cathode electron gun that is trained on the surface of a specimen. The energy of the electron beam causes many minerals or synthetic phases to produce colored light or "cathodoluminescence." This phenomenon is similar to the better-known fluorescence, but the excitation source is an electron beam rather than ultraviolet radiation, and the intensity of cathodoluminescence is stronger than that of fluorescence for a given mineral [6].

Several different types of composites and one ceramic material were sent to SMMI Microscopes for evaluation of this technique. The specimens included an uninhibited carbon-carbon (Hitco 8A), a boron-inhibited carbon-carbon (IMCC-3), a chemical vapor infiltrated carbon-carbon (D3-1A), a Nicalon/SiC composite and a ceramic specimen (NZP). Initial results did not reveal luminescence in the various materials according to SMMI; however, it is felt that a more thorough examination is necessary.

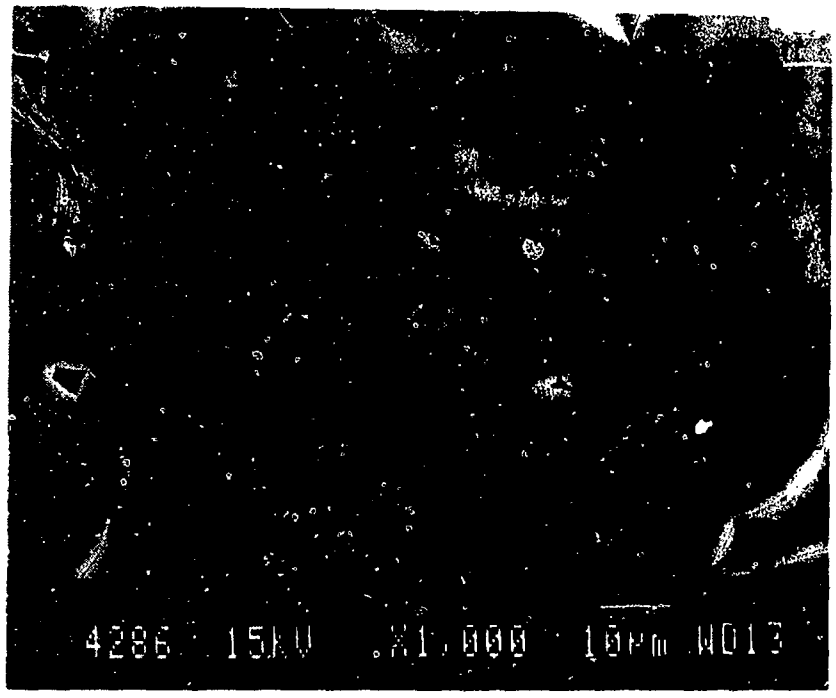
Cathodoluminescence is also a feature found on scanning electron microscopes. A couple of disadvantages with this combination are the small size of the area that can be examined (less than 50 micrometers) and the inability to directly observe CL in color.



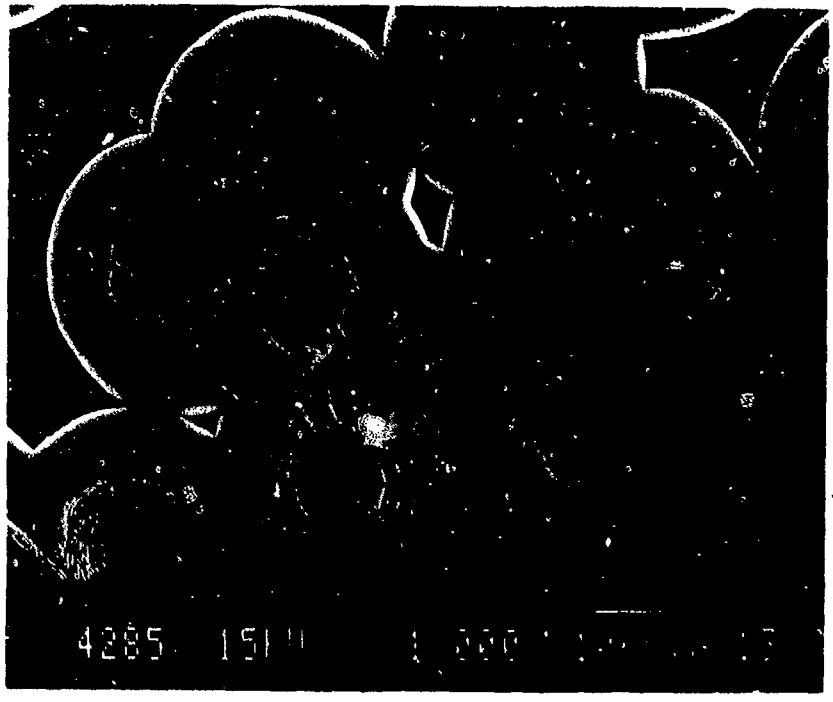
(a)

(b)

Figure 31. SEMs Showing Surface of RX715V Carbon-Carbon (2500X). (a) Pitch Fiber and Pitch Matrix, Nonetched Surface; (b) Etched Surface.



(a)



(b)

Figure 32. SEMs Showing Noveltex CVD/PAN Carbon-Carbon (1000X). (a) Nonetched Surface; (b) Etched Surface.

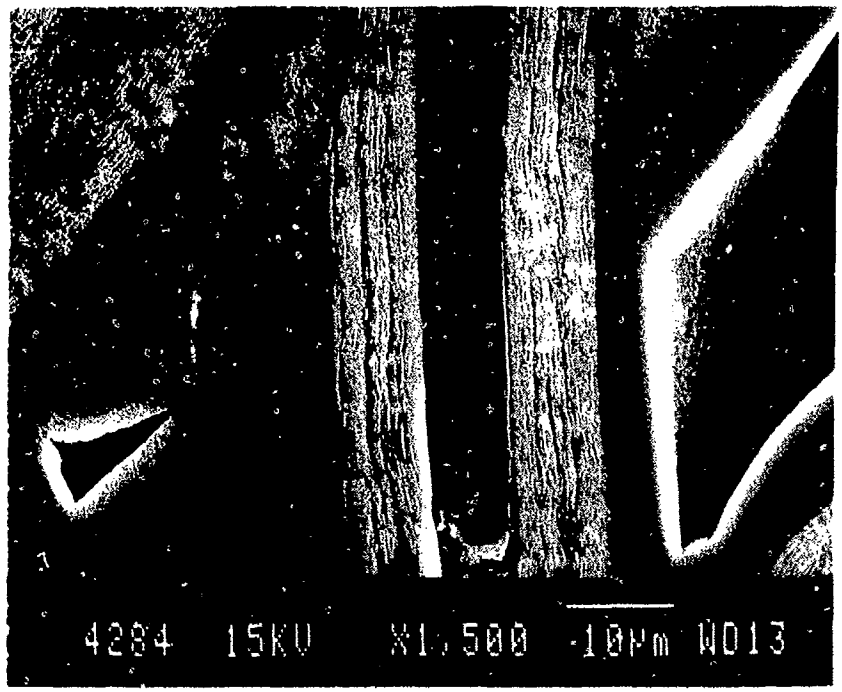


Figure 33. SEM Showing Etched Surface of PAN Fiber (1500X).

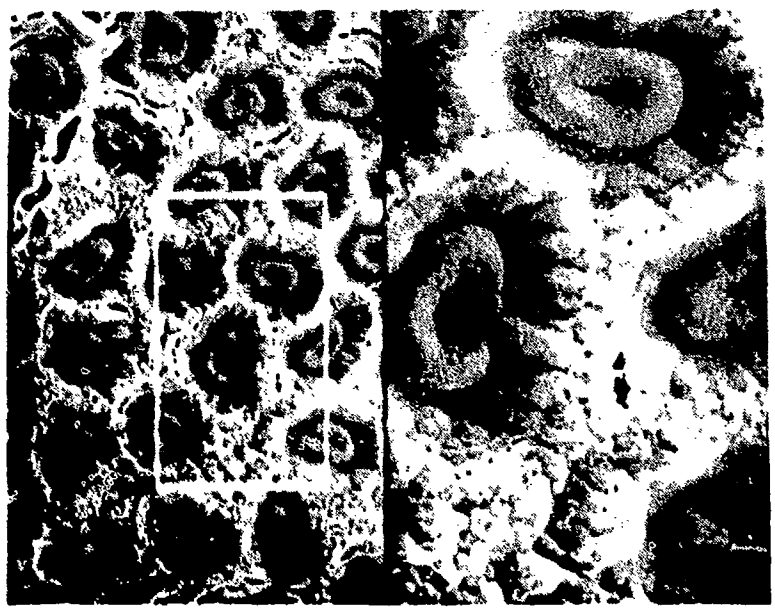


Figure 34. SEM Showing Argon-Etched Surface of Hitco 8A Carbon-Carbon (2000X).

7.5 Transmission Electron Microscopy

A Jeol 100CX transmission electron microscope (TEM) was utilized to examine carbon-carbon composites. This technique proved invaluable for investigating fiber and matrix defects, interface quality, and composition. The degree of graphitization can also be determined by utilizing X-ray diffraction in conjunction with the TEM. Carbon-carbon samples were prepared as follows. A specimen was cut with a diamond wafer saw to a thickness of approximately 0.005 inch. A 3-mm disk was prepared from the first cut with a core drill. The disk was then ion milled on both sides at 6 kV for 12 hours, with a gun tilt of approximately 15 degrees. The ion milling was accomplished on a Gatan M-600.

8. NONDESTRUCTIVE EVALUATION

8.1 Ultrasonic Evaluation

A two-dimensional carbon-carbon composite panel from Hitco Corporation was ultrasonically scanned to determine if this technique could be used to detect porosity on a per-ply basis. The pore density is similar to the Hitco 8A and 8B material. The first scans on the immersion-type ultrasonic scanning apparatus indicated that the open porosity of the panel was filling with the immersion liquid (water). The water-filled areas appear densified, thus masking the porosity. This problem was solved by applying a gel coating to the panel surfaces to prevent water infiltration. The gel coating does not attenuate the signal. A test was conducted using a 1-MHz transducer. Due to the complex weave structure and the high porosity of the panel, 50 percent of the signal was attenuated, making any determination difficult. The frequency was lowered using a 200-kHz transducer. The attenuation loss was reduced to 16 percent, but the penetration resolution was very poor. The C-scan shown in Figure 35 possibly shows density variations throughout the thickness of the panel. From these studies it was concluded that ultrasonic examinations cannot be used to detect small porosity variations on a per-ply basis, although detection of gross porosity variations and gross defects through the entire thickness is possible.

8.2 Scanning Acoustic Imaging

The scanning acoustical microscope (SAM) measures changes in ultrasonic energy as it passes through or is reflected by a specimen. Images are created by processing reflected signals taken from two-dimensional sampling at various depths. The output is claimed to be a high-resolution, high-magnification image of the internal features of the specimen produced on a monitor or CRT. The acoustical energy can penetrate virtually any material. The principle is used to detect variations in characteristics such as in grain structure, grain size, particle size, and density throughout the specimen. In the past this technique has been used to measure the presence of discontinuities such as voids, cracks, delaminations, and inclusions [7].

A number of specimens were sent to the Department of Engineering Science and Mechanics at Pennsylvania State University. This included one specimen of as-received Hitco 8A carbon-carbon composite, one Hitco 8A carbon-carbon specimen mounted and polished, and Hitco 8A11, oxidized four hours at 785°C (1445°F). The oxidized and unoxidized specimens were sent to determine if the scanning acoustical microscope could detect the differences in porosity between the two. One sample of IMCC-3 carbon-carbon containing an oxygen inhibitor in the matrix was also included to determine if the scope could detect the inhibitor.

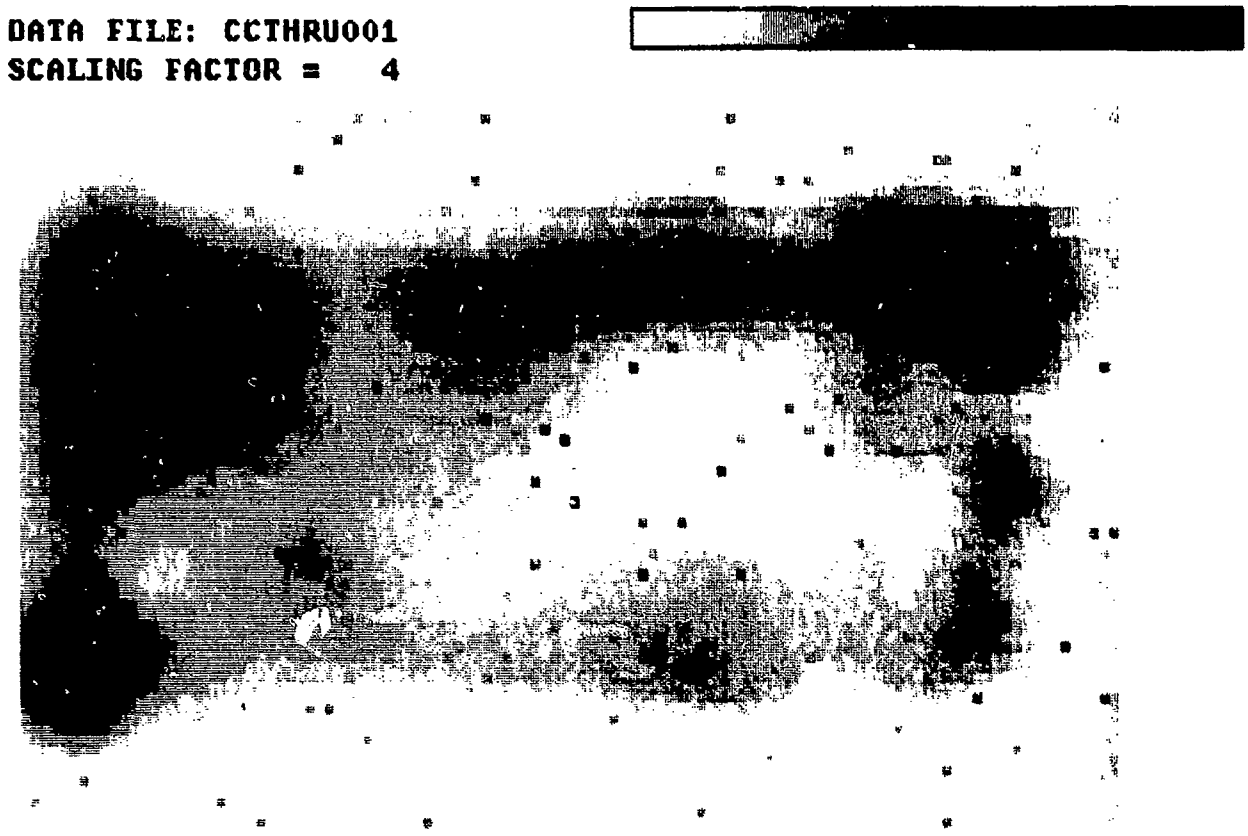


Figure 35. A 200-kHz Ultrasonic Scan of Hitco Carbon-Carbon Specimen Showing Possible Density Variations.

The acoustic scans on the Hitco 8A materials illustrated poor resolution. The 0-degree and 90-degree fibers of the polished specimen were obscured, as shown in Figure 36. The larger cracks common to this material were poorly visible. The resolution was poor on both the unoxidized and oxidized (Figure 37) 8A carbon-carbon specimens. The IMCC-3 carbon-carbon specimen containing the various inhibitors proved most interesting. The inhibitors which would be expected to be of high acoustic impedance appear as white clusters throughout the imaged area as shown in Figure 38.

In conclusion, the preliminary results indicate that poor resolution and limited depth penetration eliminate this technique as a possible tool to characterize the carbon-carbon microstructure effectively. There is some indication the technique may be used to detect the presence or distribution of oxygen inhibitors. Claims indicate 1-micron resolutions are possible; however, the depth penetration is limited even further at these parameters.

Six reticulated vitreous carbon foam specimens were also sent to determine if a three-dimensional model of the foam structure could be imaged. The specimens sent ranged from 30 pores per inch to 100 pores per inch. Three specimens were sent unaltered and three specimens were resin impregnated to fill the open porosity of the foam. It was felt that filling the open porosity may enhance the image of the foam structure. One scan of a resin-impregnated, 80 pore-per-inch foam specimen was returned. There was very little subsurface structure evident.

Another variation on the acoustical technique that may prove valuable is the scanning laser acoustical microscope (SLAM). This scope measures transmitted energy rather than reflective energy. The technique can be used to measure real-time behavior such as progressive crack growth at depths greater than the SAM can penetrate. A holographic processing technique can be combined with the SLAM to create reconstructions in three dimensions. This technique could possibly be used to create a three-dimensional model of the carbon foam. Specimens were sent to Sonoscan Inc., a supplier of acoustical microscopes, for evaluation. However, repeated attempts to have these specimens evaluated and returned were unsuccessful.

8.3 Computer Tomography

A Hitco carbon-carbon composite panel was used to evaluate laminography/dual energy (LAMDE) computer tomography (CT). Computed tomography is the reconstruction by computer of a tomographic plane or slice of an object. An object is placed between a collimated X-ray fan beam and an X-ray sensitive detector. X-ray intensity measurements are made by the

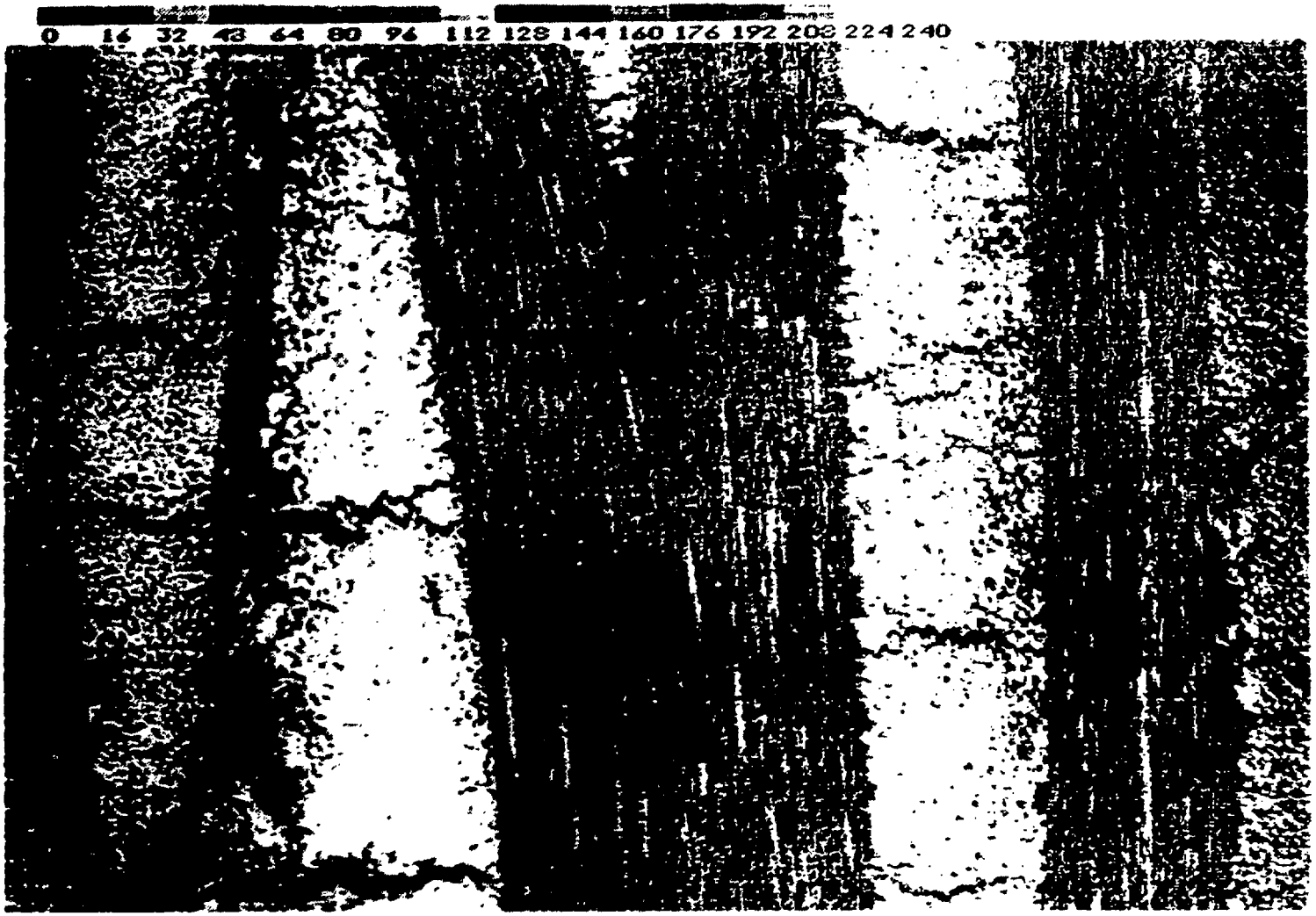


Figure 36. Scanning Acoustic Micrograph, 400 MHz, 1-mm Scan Width of Polished Hitco Carbon-Carbon Specimen.

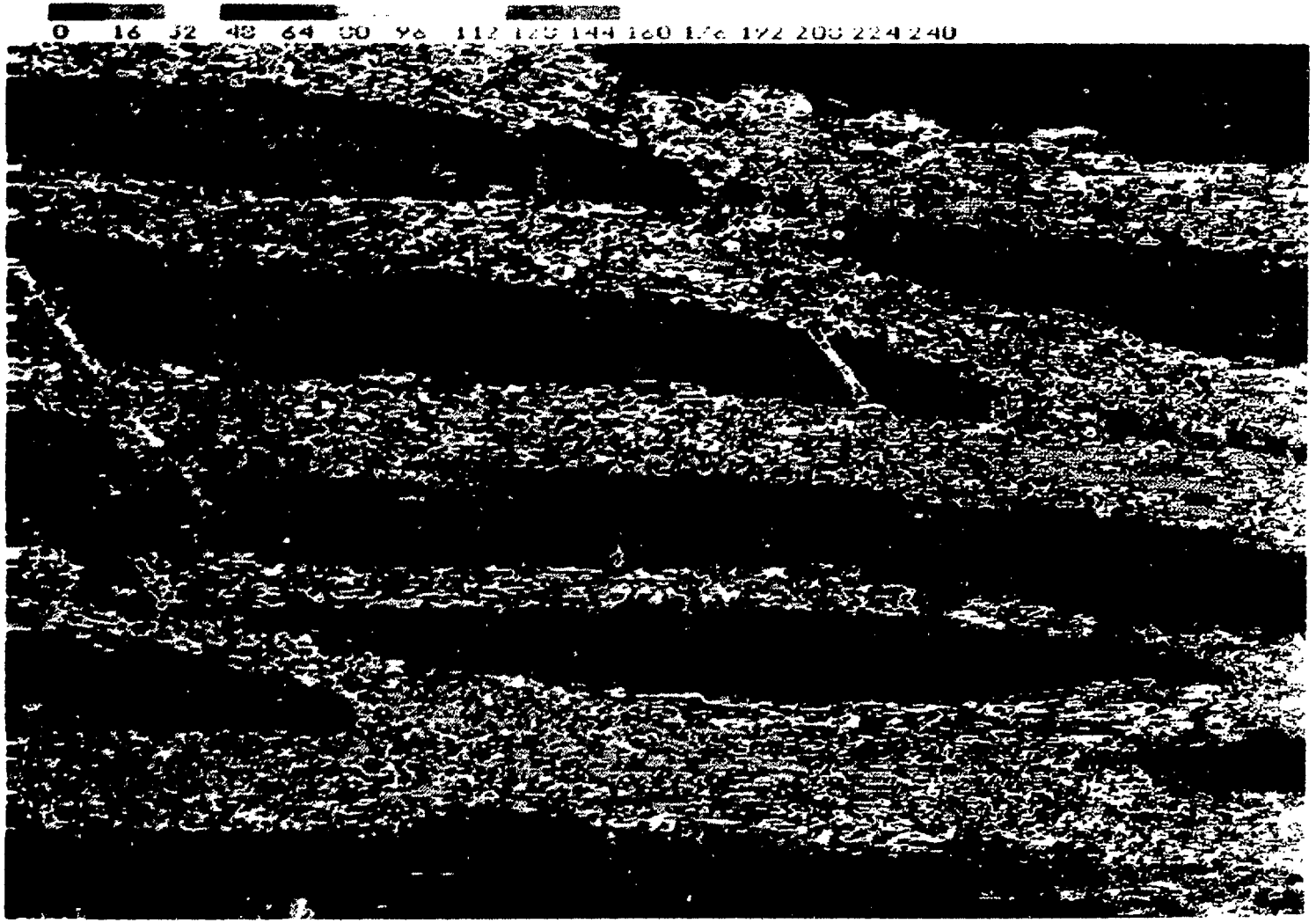


Figure 37. Scanning Acoustic Micrograph, 200 MHz, 2-mm, Oxidized Hitco Carbon-Carbon Specimen.

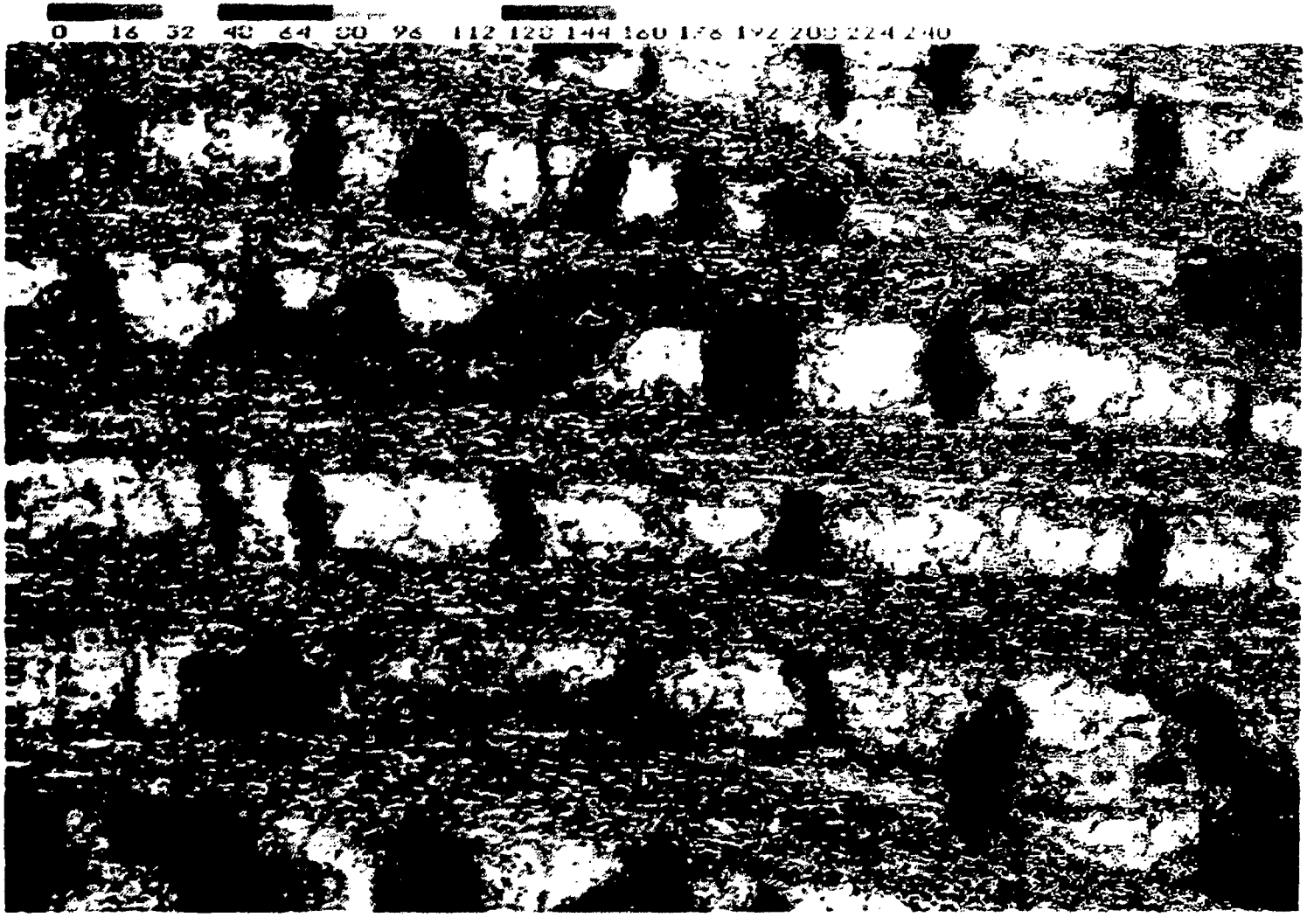


Figure 38. Scanning Acoustic Micrograph, 200 MHz, 2-mm, IMCC-3 Carbon-Carbon Specimen with Oxygen Inhibitors.

detector as the object is fully rotated within the X-ray fan beam. A cross-sectional image is then computer generated from the data to provide a highly-accurate internal view of that slice (Figure 39). LAMDE is a second-generation translate-rotate CT machine. There are 144 detectors in the detector package, and the turntable can support objects up to 600 mm in diameter and 250 pounds. The apertures are variable and automatic. The resolution aperture varies from 1.2 mm to 4.5 mm, and the source aperture varies from 1.2 mm to 15 mm. Control of the apertures and the specific system configuration allows for an optimum spatial resolution of 250 microns [8]. Past CT investigations of carbon-carbon composites allowed detailed mapping of high- and low-density areas within the interior and the imaging of multilayered coatings.

As with the ultrasonic evaluation, the goal was to detect and record the porosity in carbon-carbon composites on a per-ply basis. If this could be accomplished, the next step would be to image microstructural damage produced by thermal cycling. Interpretation of the CT scans proved difficult, since there was no established baseline to compare them with. It would be beneficial to scan a carbon-carbon composite with known defects or deliberate inclusions to establish baseline operational parameters. Possible density variations were detected (Figure 40) in the carbon-carbon panel scanned, but the spatial resolution is not sensitive enough to detect voids and microcracking on a per-ply basis. A new-generation, high-resolution CT system, designated the Tomoscope, has recently become available for use. This unit can achieve spatial resolutions of 25 microns or better, which is an order of magnitude better than conventional CT systems. The system can inspect objects up to 100 mm in diameter [9]. Once the unit is operational, carbon-carbon specimens will be evaluated. CT is still in the early stages of development. The technique has future potential for nondestructively evaluating the microstructure of complex reinforced materials such as carbon-carbon.

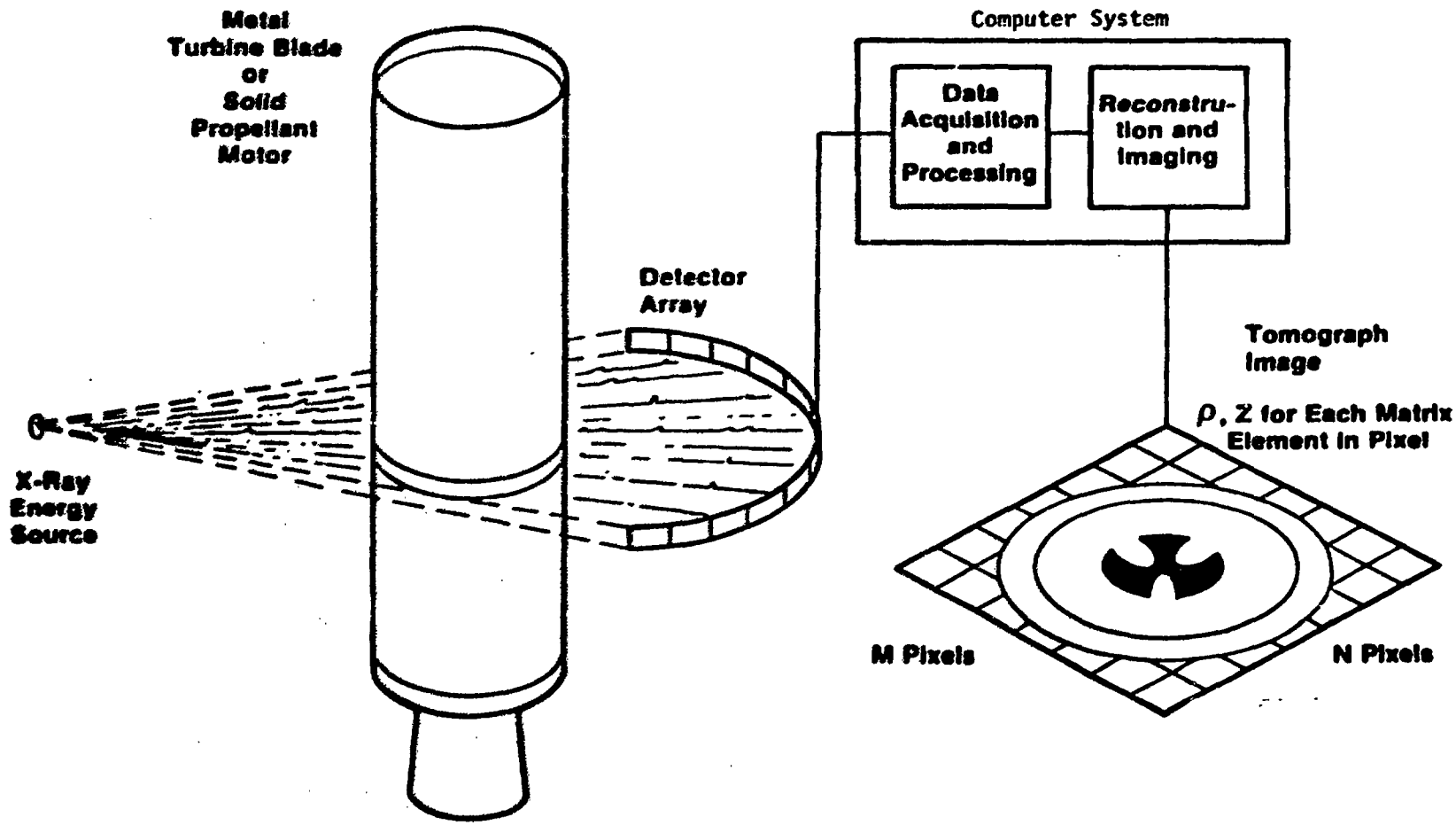


Figure 39. Laminography/Dual Energy CT Principle of Operation.

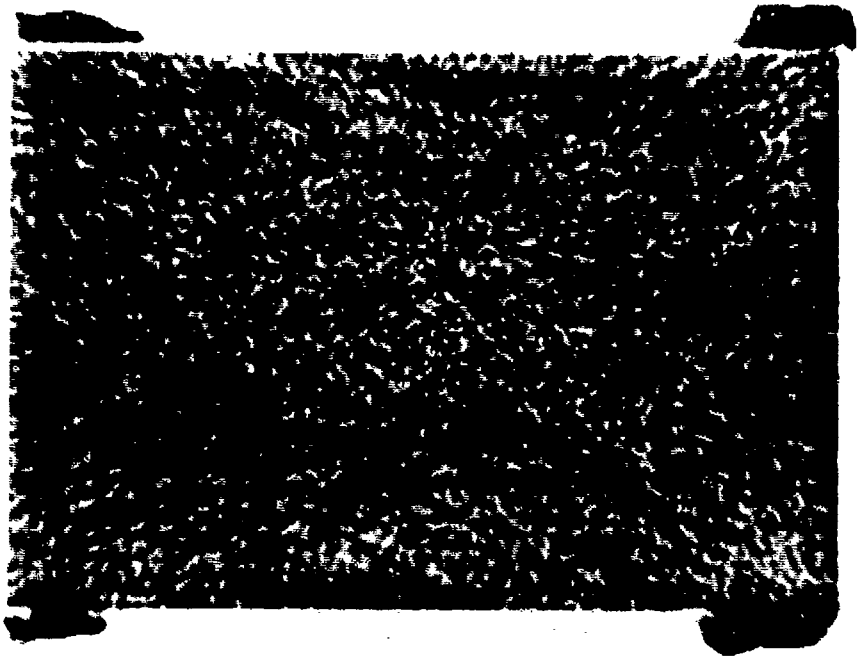


Figure 40. Computer Tomography Scan of Hitco Carbon-Carbon Showing Possible Density Variations.

9. CONCLUSIONS

Characterizing carbon-carbon composites can be challenging for several reasons. The high porosity and mismatch in properties of the various constituents complicates characterization techniques. Further complications surface if the material has become fragile due to oxidation or mechanical testing prior to characterization. These variables will influence the type of specimen preparation chosen for proper characterization.

Vacuum impregnating the open porosity with an epoxy resin was found to be most important to preserve intact the internal microstructure during subsequent preparation procedures. If the porosity resists standard impregnation techniques, then pressure may carefully be applied to force the infiltrating resin into the smallest pores. A fluorescent dye can be added to the impregnation resin to enhance minute cracks or pores during microscopic examination.

All sectioning techniques will damage the constituents on the surface of the composite specimen. The depth of this damage was not determined. If subsurface damage is a concern, the only recourse is to carefully remove additional damaged material using grinding and polishing techniques. A versatile saw for sectioning carbon-carbon composites should have a low speed capability and employ a thin diamond impregnated blade. The cooling fluid of choice is distilled water, but in some cases, such as when water soluble inhibitors are present, an oil-based coolant may be used.

Several mounting or plugging options are discussed in this report. The most important rule is to try and match the hardness and abrasion resistance of the specimen with that of the mounting medium. It should also be determined if methods that use heat or pressure will damage the specimen.

Polishing techniques vary greatly. By following a few basic ground rules outlined in this report, the development of a polishing technique for a specific material should not be difficult. Automated equipment tends to provide the best results, since human error is kept to a minimum. As stated earlier, experience, common sense, and trial and error will dictate the best approach for a specific application.

Optical microscopy may be used to examine composite quality. A scope equipped with polarization and fluorescence detection capabilities is a valuable tool. Darkfield illumination has limited capabilities. The technique was used to highlight fluorescent-tagged impregnation resin when a fluorescent scope was unavailable.

The scanning electron microscope is an invaluable tool for microscopic characterization. The compositional mode, which detects atomic number contrast is most useful when examining at inhibited carbon-carbon. The backscattering mode may be used to enhance the different layers of oxidation inhibitors on coated carbon-carbon composites. Ion bombardment or cathodic vacuum etching can also be used to produce structural contrast by selective removal of atoms from the specimen surface. This can reveal microstructural surface features of the various constituents in the SEM. The transmission electron microscope is a useful tool for microexaminations of fiber/matrix defects, interface quality and compositional analysis.

Nondestructive evaluation techniques such as ultrasonic scanning and computer tomography (at this time) are limited to detection of macroscopic damage and gross porosity variations through the entire thickness. Spatial resolution needed to detect porosity in carbon-carbon composites on a ply-to-ply basis is not available at this time. Scanning acoustic imaging also demonstrated poor resolution and has limited depth penetration. The technique did seem to enhance inhibitors within the matrix, indicating that with further work the method could be developed to qualitatively and quantitatively evaluate the inhibitors.

Technological advances of existing characterization techniques and the development of new characterization techniques are occurring rapidly. It is well worth the time to stay abreast of these advances.

REFERENCES

1. H. Brent Carroll, "Advanced Carbon-Carbon Involute Exit Cone Program, Volume 1," U.S. Air Force Technical Report, AFWAL-TR-86-4096 (November 1986).

2. "Photography through the Microscope," Kodak Publication P-2 Cat 152-8371 (1980).

3. L. L. Clements, "Fiber Composite Materials," in Metals Handbook, Ninth Edition, Volume 9, Metallography and Microstructures (1985).

4. "The Sarastro Story," in Microscope Technology and News, Volume 1, Number 10 (October 1989).

5. G. Petzow, "Metallographic Etching," in Buehler, Inc. publication (1976).

6. R. D. Hagni and M. Karakus, "Cathodoluminescence Microscopy: A Valuable Technique for Studying Ceramic Materials," in MRS Bulletin, Volume XIV, Number 11 (November 1989).

7. J. C. Bittence, "Greater Precision for Materials Analysis," Advanced Materials and Processes, Volume 136, Issue 5 (November 1989).

8. Wright Laboratory Materials Directorate X-ray Computed Tomography Research Facility Bulletin, Tomoscope CT System (June 1990).

9. Wright Laboratory Materials Directorate X-ray Computed Tomography Research Facility Bulletin, LAMDE CT System (June 1990).

*U.S. GOVERNMENT PRINTING OFFICE: 1992-648-127/62471 Region 5.